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TECHNICAL REPORT  
69-57-FL

STUDY OF EXTRACTABLE SUBSTANCES AND MICROBIAL PENETRATION OF POLYMERIC  
PACKAGING MATERIALS TO DEVELOP FLEXIBLE PLASTIC CONTAINERS FOR  
RADIATION STERILIZED FOODS

by

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U. S. ARMY NATICK LABORATORIES  
Natick, Massachusetts 01760

## FOREWORD

The fact that a food has been sterilized is no guarantee that it will not be recontaminated. For this reason alone, finding the right packaging for radiation-sterilized foods is an objective of greatest importance, and its attainment is an essential factor in moving irradiated foods into commercial use.

The Technical Report FD-3 on "Extractives and Functional Performance of Flexible Packaging Materials for Use in Radiation Sterilization of Prepackaged Foods," December 1964, presented the initial research performed by the Continental Can Company, Inc. under Contract No. DA19-129-AMC-162(N). The work covered in this report, also performed by Continental Can Company, Inc., under the same contract, represents an investigation of the development of flexible lightweight containers capable of withstanding rough handling and storage, retaining protective qualities during storage - and this without any adverse effects on the food contained therein. Mr. G. O. Payne, Jr. was the Project Leader; Mr. C. J. Spiegl, the Principal Investigator - Phase I; Mr. F. E. Long, the Principal Investigator - Phase II; and W. C. Dougherty, B. H. Ellis, R. J. Hansen, R. M. Jackson, F. J. Kraus, D. T. Maumder, J. E. Moreau, M. R. McKee, C. C. Schmiede, and C. F. Schmidt, the collaborators in the research work, for Continental Can Company, Inc.

The U. S. Army Natick Laboratories Project Officer was Mr. J. J. Killoran, of the Engineering Science Division, General Equipment and Packaging Laboratory and the Alternate Project Officer was Dr. Eugen Wierbicki, of the Irradiated Food Products Division, Food Laboratory.

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### ABSTRACT

Flexible packaging laminates were developed and evaluated for use with prepackaged irradiation-sterilized ham, bacon and chicken. Laminate structures comprised poly (ethylene terephthalate) as the outside ply, aluminum foil as the middle ply, and heat sealable inside ply, namely film of polyolefin, polyamide or polyester. These laminates were evaluated for food compatibility, low-temperature resistance, and resistance to microbial and insect penetration. The flexible laminate with the polyamide as the food-contacting film was found satisfactory for use with prepackaged ham, chicken and bacon in various test environments over a one year storage. A flexing apparatus, developed to induce bacterial penetration through defects in flexible packages, was found capable of detecting leaks not visible to the naked eye. The three types of flexible laminates showed no negligible weight loss and no package rupture in shipping vibration tests performed as low as  $-180^{\circ}\text{C}$ . Exposure of the flexible laminates to three types of boring insects over a three month period caused no complete penetration of the packages under study.

## PHASE I

### 1. PROJECT OBJECTIVE

To assist the U. S. Army Natick Laboratories in developing information necessary for specifying the types of flexible packages suitable for military feeding in which the contained foods are sterilized by ionizing radiation after packaging.

### 2. SCOPE

To determine the nature and amount of extractives released into food simulating solvents contained in flexible packaging materials subjected to sterilizing doses of ionizing radiation. The results are to be used by the U. S. Army Natick Laboratories in support of one or more petitions to the Food and Drug Administration for obtaining their acceptance of these materials under the Federal Food, Drug and Cosmetic Act for in-package radiation sterilization of foods.

### 3. PROGRAM OF WORK

The nature and concentration of extractives from six (6) irradiated flexible packaging materials was determined by chemical and microanalytical techniques and compared with extractives from the same material not irradiated but held in contact with the solvent under similar conditions of storage. In addition to a study of extractives, which is the primary function of Phase I, certain supplementary studies were undertaken.

These included:

a. Subjective evaluation of the effects of irradiation on packaging materials, including physical changes, odor and color.

b. Observation of functional performances.

c. Analyses of the major components of head space gases in polyethylene pouches filled with water, irradiated and stored for one month.

d. Experiments, as required, to elucidate extractability data.

The six (6) widely used and commercially available food contacting films that have FDA approval for other food packaging uses and which were selected as the most likely to fulfill the functional requirements for food contact use for irradiated foods are:

Polyolefin

Polyvinylidene Chloride Copolymer

Polyvinyl Chloride

Polystyrene

Polyester

Polyamide

In selecting polymers to form the food-contacting films, it was determined that they should not contain any intentional additives that might confuse the results of polymer extractability and secondly, that the film thicknesses should be uniform in order to allow comparisons between materials. To that end, the food-contacting films were all of one mil thickness and without plasticizers even though these conditions are not necessarily the preferred commercial practice. In the case of polyethylene, a 3 mil film was also studied.

#### 4. SUMMARY OF RESULTS

Completion of Phase I of the contract has shown that no significant change occurs in extractability of six classes of polymeric material in contact with food simulating solvents during irradiation at food sterilization doses. Observation of functional performance of the flexible laminate pouches as extractive test cells indicates that the preferred food contacting material classes for further study in Phase II are polyester, polyamide, and polyolefin.

The final report of the Phase I effort has been issued by the U. S. Army.<sup>1</sup> Food Additive Petition 5M-1645 has been filed by the U. S. Army to amend existing Food and Drug Administration laws to permit the use of certain food contacting polymer films for radiation sterilization of pre-packaged foods.<sup>2</sup>

#### 5. RECOMMENDATIONS

No significant change in extractability was found in six classes of polymeric materials at food sterilization radiation doses. Therefore, the recommendations of food contacting materials to be investigated in the Phase II pouch development and pouch performance evaluations should be guided by the observed functional performance, as well as established

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<sup>1</sup> Technical Report FD-3, Extractives and Functional Performance of Flexible Packaging Materials for Use in Radiation Sterilization of Pre-packaged Foods. G. O. Payne, Jr., C. J. Spiegl, December 6, 1964, U. S. Army Material Command, U. S. Army Natick Laboratories, AD455946.

<sup>2</sup> "Packaging Materials for Use in Radiation Preservation of Pre-packaged Foods," Amendment to the Paragraph 121.2543. "Department of the Army-Notice of Filing of Petition for Food Additives Packaging Materials for Use in Radiation Preservation of Pre-packaged Foods." 13 July 1965.

flexible packaging technology. Since the actual foods to be used in contact with the flexible pouch studies in Phase II will be high protein foods (meats), the preferred food contacting polymer types would be polyester, polyamide, and polyolefin.

## PHASE VI

### 6. OBJECTIVE

To assist the U. S. Army Natick Laboratories in developing information necessary for specifying the types of flexible packages suitable for military feeding in which the contained foods are sterilized by ionizing radiation after packaging.

### 7. SCOPE

To develop a limited number of suitable flexible packages for certain radiation sterilized foods and the additional necessary protective jackets and shipping cartons so that the results may be used in establishing military purchase specifications for appropriate package systems.

### 8. PROGRAM OF WORK

Three single service (4 to 5 oz.) flexible packages systems were developed for evaluation with pre-packaged radiation sterilized (4.5 megarad-minimum dose) ham, chicken, and bacon.

Following the development and fabrication of the flexible packaging systems, functional performance, i.e., for military feeding, of the three packaging systems was determined in a series of specific studies which are reported in detail in the sections noted in Figure 1.

#### 8.1 Selection of Foods to be Packaged

Three foods, ham, chicken, and bacon, were selected by the Project Officer, U. S. Army Natick Laboratories, as the food items for which flexible packages were most desired in their program.

## 8.2 Selection of Types of Food Contacting Polymer Films for Package Development

Based upon the results of Phase I experiments, the preferred food contacting appropriate for further package development studies were polyamides, polyesters, and polyolefins. However, additional polymer films were included for evaluation in Phase II.

## 8.3 Selection of Laminate Structure for Pouch Development

Using the information developed in Phase I and in conjunction with the Project Officer, U. S. Army Natick Laboratories, the following laminate structure was selected for the flexible pouch development and performance evaluation studies:

<u>Position in Laminate Structure</u>	<u>Material</u>
Food Contacting Films	Polyamide, polyester, polyolefin
Adhesive-Primer or extrusion lamination dependent on food contacting film type	L-4533, epoxy type (app. 0.5 lbs/1000 sq. ft.)
Middle Ply	Aluminum Foil (0.5 mil)
Adhesive Primer	L-4533, epoxy type (app. 0.5 lbs/1000 sq. ft.)
Outside Film	Polyester* (0.5 mil)

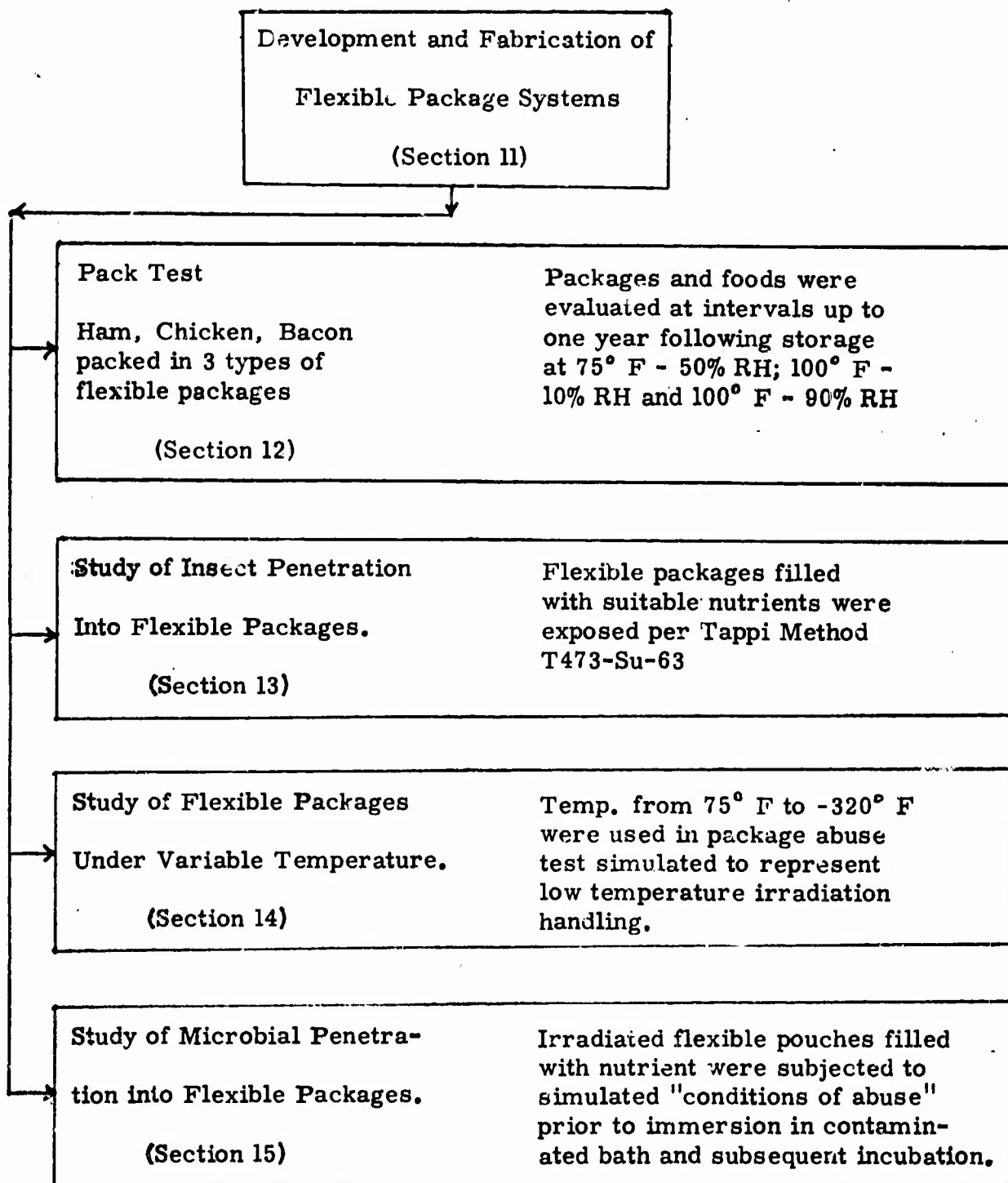
\* Mylar "A" - Supplier: E. I. DuPont de Nemours

## 8.4 Flexible Pouch Construction

The dimensional specifications of the flexible pouches were as follows:

FIGURE 1

**PHASE II - FLOW DIAGRAM OF PROGRAM OF WORK**



Volume: App. 5 fluid ounces.

Dimensions: 4-1/2" x 7"  $\pm$  1/16" in either or both directions with seals 3/8" wide on the sides. The opening shall be on the edge of the smaller dimension.

#### 8.5 Flexible Packaging System Components

The single service flexible package consists of a laminated pouch enclosed in a protective paperboard jacket. For many of the studies corrugated cartons were required for irradiation, shipping, and testing. The specifications for these packaging components are as follows:

##### Protective Jackets

The protective paperboard jackets were fabricated from 16-point solid sulfate board. Glue was applied to the inside rectangular surface of the protective jacket prior to inserting the flexible pouch and closing of the jacket. The use of this glue minimized the shifting of the pouch, resulting in enhanced durability of the flexible pouch in actual use. (See Figures 2 and 3.)

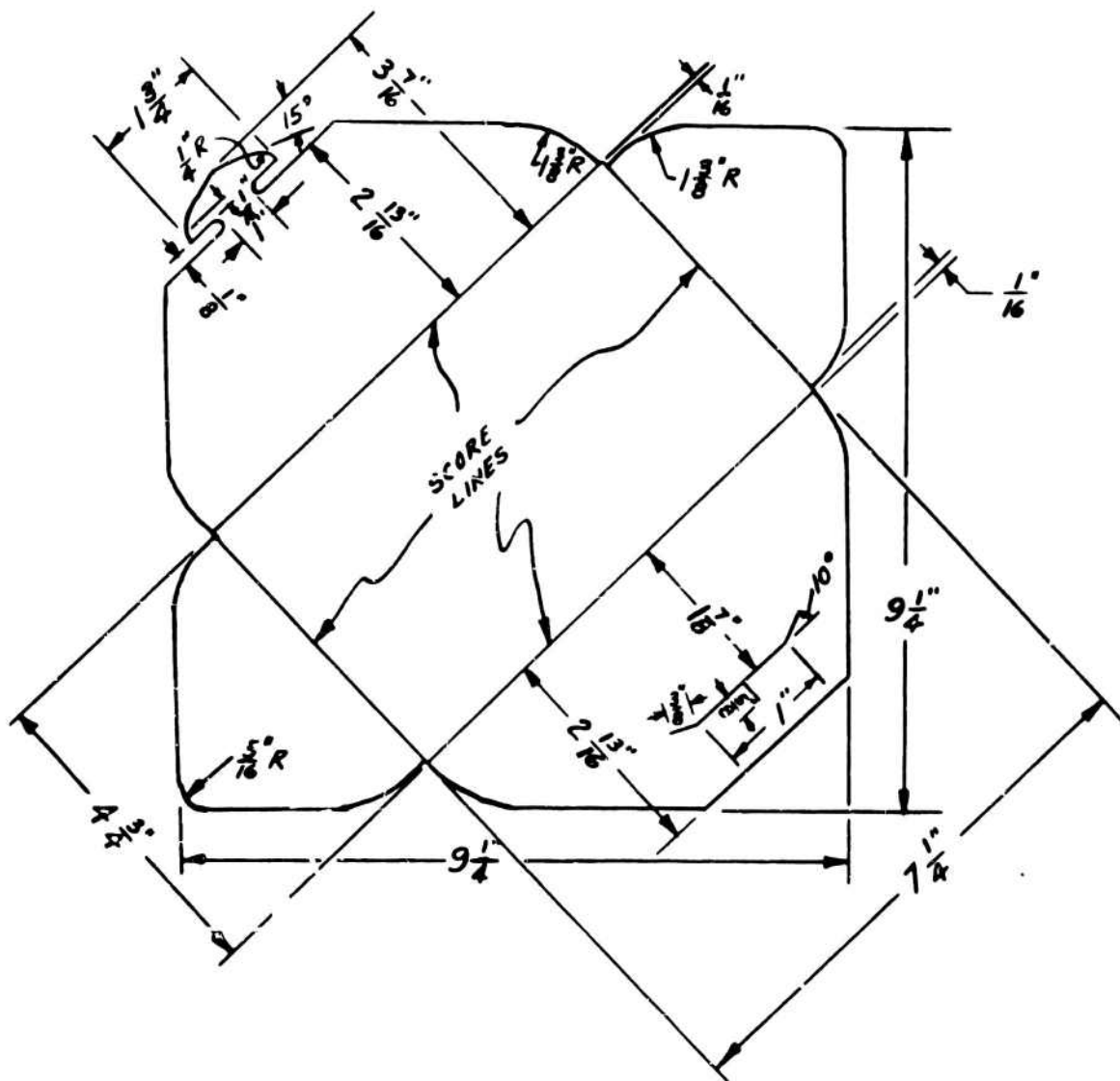
##### Inner Corrugated Carton

Corrugated paperboard inner shipping cartons were designed and fabricated to contain 48 pouches. The dimensions of the inner shipping carton (as shown below) were selected to obtain the most uniform radiation dose distribution in the carton when irradiated in the U. S. Army Natick Laboratories cobalt 60 source.

##### Material:

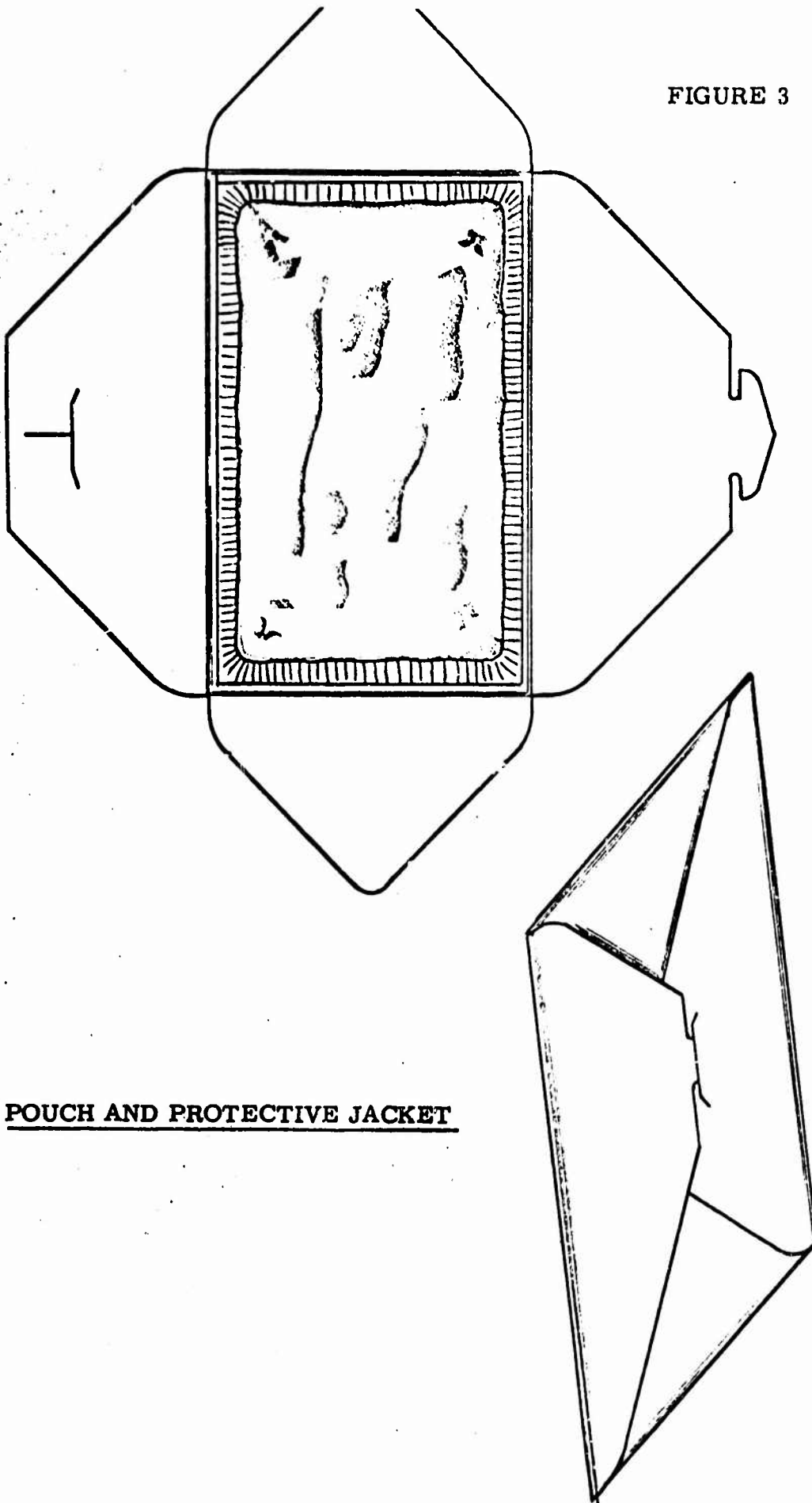
Type I, Class 1, Style  
RSC of Specification  
PPP B-636 except bursting  
strength of not  
less than 200 p.s.i.

FIGURE 2



PROTECTIVE JACKET

FIGURE 3



POUCH AND PROTECTIVE JACKET

<u>Inside Dimensions:</u>	15" L x 12-1/2" W x 4-3/4" D
<u>Wall Thickness:</u>	App. 1/8"
<u>Outside Dimensions:</u>	15-1/2" L x 12-5/8" W x 5-1/2" D
<u>Divider Dimensions:</u>	4-3/4" W x 15" L
<u>No. of Pouches per Carton:</u>	48 (2 layers of 24 each)
<u>Total Volume of Pouches:</u>	App. 240 fluid ounces

#### Packaging Configuration

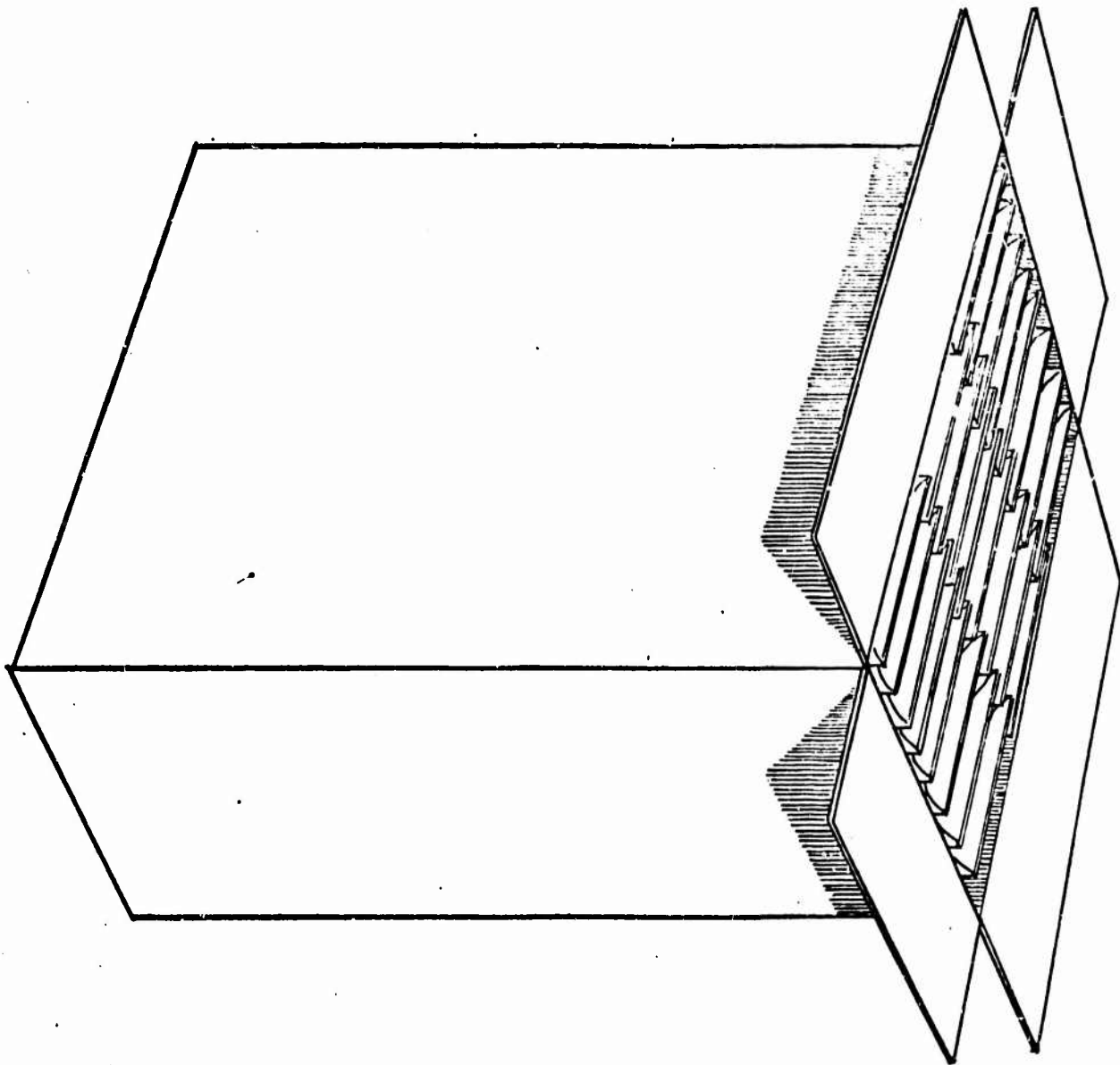
Figure 4 shows the cartons and the method of packing the pouches. The overlapping system of pouch arrangement gives a closer packing and partially eliminates some of the air spaces; this is advantageous both for shipping and for radiation efficiency.

#### Outer Shipping Cartons

Outer shipping cartons were fabricated to hold two inner cartons as shown in Figure 5. The carton specifications were as follows:

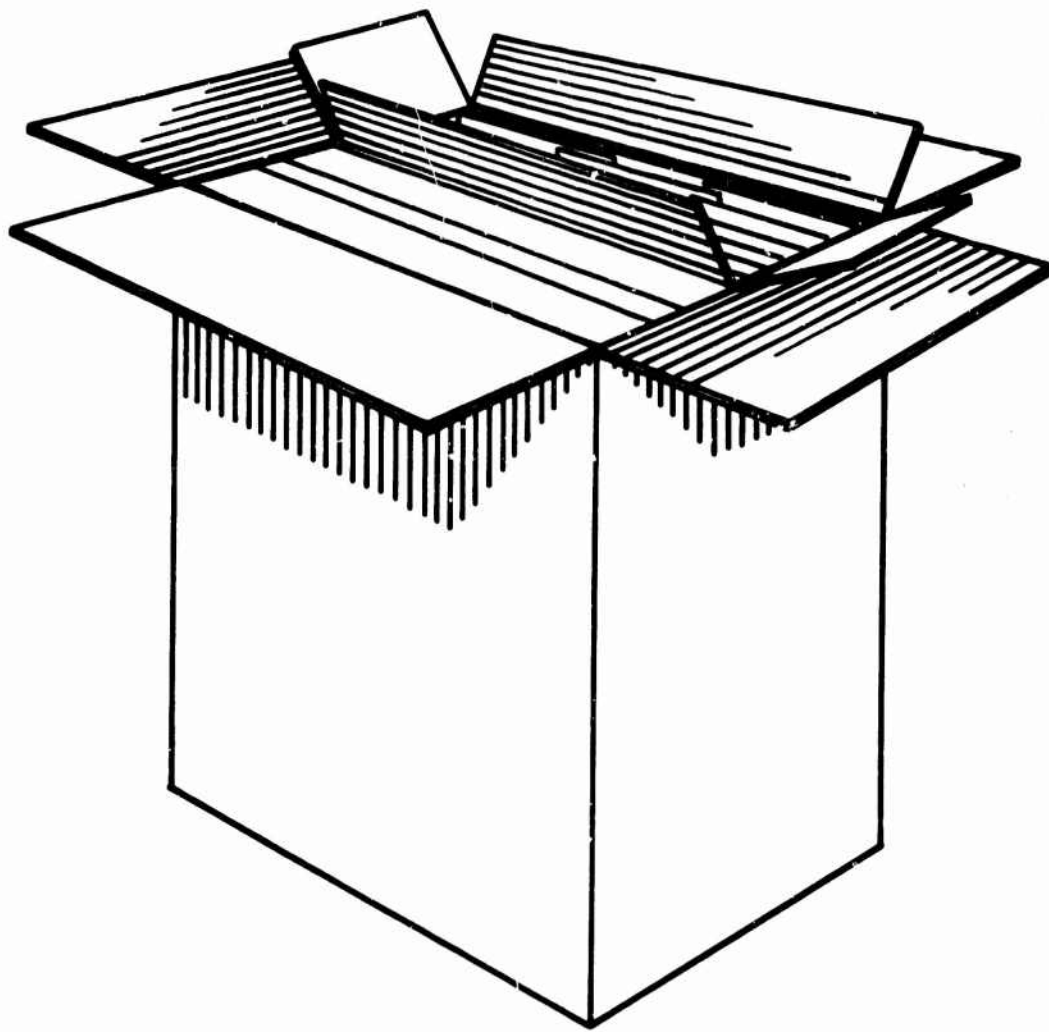
<u>Material:</u>	Type I, Class 1, Style RSC of Specification PPP B-636 except bursting strength of not less than 200 p.s.i.
<u>Inside Dimensions:</u>	15-5/8" L x 12-3/4" W x 10-5/8" D
<u>Wall Thickness:</u>	App. 1/8"
<u>Outside Dimensions:</u>	15-7/8" L x 13" W x 11-1/8" D
<u>No. of Inner Cartons:</u>	2
<u>No. of Pouches per Shipper:</u>	96

FIGURE 4



INNER CORRUGATED CARTON

**FIGURE 5**



**OUTER CORRUGATED SHIPPER**

## 9. SUMMARY OF RESULTS

### Development and Fabrication of Flexible Packages

Twenty commercially available polyamide, polyolefin, polyester, polyvinyl chloride, polyvinylidene chloride, polystyrene, and polycarbonate polymeric films were screened as possible food contacting films in flexible laminate pouch structures for radiation sterilized foods. Based on the results of the screening study three films, Marlex 6050, Nylon 11, and Vitel 409, were selected as having the most promising combination of properties for food contacting films (See Section 11).

The three films were used to develop and fabricate flexible packages, all using the same base lamination, protective jackets, and shipping containers, for extensive testing in the four additional studies shown in Figure 1.

### Performance of Flexible Packages - General

The studies of insect penetration, low temperature behavior, and microbial penetration were undertaken to demonstrate the general feasibility of using flexible packages for radiation sterilized foods, whereas the pack tests, which are summarized in sections 9.1, 9.2, 9.3, were evaluations of packaging performance of radiation sterilized hams, chicken, and bacon in specific flexible packages.

Insect penetration into flexible package structures of the type used in this project is not a factor of concern in package performance. (See Section 13 for detailed report.)

For those food products in which the pre-packaged food must be held at extremely low temperatures during the irradiation cycle, flexible packages can be successfully used if reasonable care is exercised to minimize shock during the low temperature cycle (See Section 14 for detailed report).

Flexible package integrity was evaluated by determining the extent of microbial penetration into packages which had been subjected to a handling abuse cycle, arbitrarily defined as equivalent to military field usage. Of the three types of flexible packages tested, the flexible pouch, using Nylon 11 as the food contacting film, was the only package type that approached the level of durability of the R-2 retort type flexible package used as a comparative package. The R-2 package had undergone testing previously by the U. S. Army. In the integrity testing conducted in this project, the R-2 packages had a 10% failure rate and the Nylon 11 type pouch, developed in the project, had a 17% failure rate. In assessing the failure rate of these two types of packages in this particular test, three factors must be considered; firstly, the handling abuse cycle was extremely severe and may exceed "typical" military handling abuse which requires additional field testing to establish "standards of performance" for flexible packages. Secondly, in order to evaluate the effectiveness of supporting the seal area juncture, to enhance durability, the Vitel 409 and Marlex 6050 packages were selected for testing that design feature change and were compared to the unsupported Nylon 11 and R-2 packages. The addition of seal support to these

latter type packages will undoubtedly increase their durability (See Section 15 for detailed report.) Thirdly, increasing the thickness of the Nylon 11 film to 3 mils from 2 mils should enhance the durability of the package comparable to the R-2 package.

The tests of packs of ham, chicken, and bacon, which are summarized below, show that flexible packages are feasible containers for specific radiation sterilized foods for military usage.

#### 9.1 Single Service Flexible Packages for Ham

Of the three flexible packages extensively studied, the flexible structure using Nylon 11 as the food contacting film is the only material that can be recommended for use with ham, based upon the pack test results (Section 12).

This material is the only one that maintained adequate seal and bond strength and lack of delamination in all three test environments for 12 months storage as shown below:

#### Bond Strength\*

(Body Areas between Aluminum Foil and Food Contacting Film)

<u>Storage Environment</u>	<u>Type of Package</u>		
	<u>Marlex 6050 Laminate</u>	<u>Nylon 11 Laminate</u>	<u>Vitel 409 Laminate</u>
77° F, 50% RH	360	350	**
100° F, 10% RH	127	270	**
100° F, 90% RH	0	280	**

\* grams/linear inch-width

\*\* too brittle for measurement

Bond Strength\*

(Seal Areas between Aluminum Foil and Food Contacting Film)

<u>Storage Environment</u>	<u>Type of Package</u>		
	<u>Marlex 6050 Laminate</u>	<u>Nylon 11 Laminate</u>	<u>Vitel 409 Laminate</u>
77° F, 50% RH	410	377	**
100° F, 10% RH	233	312	**
100° F, 90% RH	0	306	**

Seal Strength\*

77° F, 50% RH	4300	4667	3733
100° F, 10% RH	4233	4167	1267
100° F, 90% RH	4200	4500	633

\* grams/linear inch-width

\*\* too brittle for measurement

All three types of flexible packages performed adequately, with exception noted below, in maintaining their barrier properties for the ham as evidenced by the summary shown below:

Per Cent Product Weight Loss After 12 Months Storage

<u>Storage Environment</u>	<u>Type of Package</u>		
	<u>Marlex 6050 Laminate</u>	<u>Nylon 11 Laminate</u>	<u>Vitel 409 Laminate</u>
77° F, 50% RH	0.10	0.09	0.07
100° F, 10% RH	0.10	0.10	0.05
100° F, 90% RH	0.10	0.10	0.10

Per Cent Oxygen in Package Head Space Gas

<u>Storage Environment</u>	<u>Type of Package</u>		
	<u>Marlex 6050 Laminate</u>	<u>Nylon 11 Laminate</u>	<u>Vitel 409 Laminate</u>
77° F, 50% RH	1.27	1.23	1.30
100° F, 10% RH	2.67*	1.15	1.20
100° F, 90% RH	1.13	1.13	1.35

\* Probably due to loss of bond between aluminum foil and the food contacting film.

The sensory contributions of the flexible packages to the food as compared to the "canned" control is negligible with the exception of the casein base adhesive used to adhere the jackets to the pouches. Unfortunately, this material was not screened for off-odor development prior to test packing. (See Section 12.1, 12.2, and 12.3 for pack test details.)

We conclude that a flexible laminate pouch structure using 0.5 mil Mylar - 0.5 mil aluminum foil - 2 mil Nylon 11 with the pouch seal area supported in the protective jacket or by increasing the thickness of the Nylon 11 to 3 mil using the pouch center glued to the protective jacket will provide a satisfactory single service flexible package for irradiated ham.

9.2 Single Service Flexible Packages for Chicken

Of the three flexible packages extensively studied, the flexible structure using Nylon 11 as the food contacting film is the recommended film for chicken. However, a linear polyethylene film could

be utilized as the food contacting film based upon its overall performance, if the polymer was modified to withstand the abuse sustained in military usage.

As shown below, the Marlex 6050 and Nylon 11 maintained adequate seal and bond strength in all environments for the twelve month storage test:

Bond Strength\*

(Body Areas between Aluminum Foil and Food Contacting Film)

<u>Storage Environment</u>	<u>Type of Package</u>		
	<u>Marlex 6050 Laminate</u>	<u>Nylon 11 Laminate</u>	<u>Vitel 409 Laminate</u>
77° F, 50% RH	393	330	633
100° F, 10% RH	217	317	337
100° F, 90% RH	160	237	387

Bond Strength\*

(Seal Areas between Aluminum Foil and Food Contacting Film)

77° F, 50% RH	435	380	663
100° F, 10% RH	350	357	**
100° F, 90% RH	203	273	393

Seal Strength\*

77° F, 50% RH	4900	4233	4167
100° F, 10% RH	5133	4533	2433
100° F, 90% RH	4900	4133	1370

\* grams/linear inch-width

\*\* Vitel 409 film too brittle to permit bond determination

### Head Space Gas Analysis in Can Control

	CO <sub>2</sub>	H <sub>2</sub>	C+A <sub>2</sub>	N <sub>2</sub>	CO	CH <sub>4</sub>	Total Head Space Volume	Vacuum In Can
77° F, 50% RH	7.1	35.2	0.8	54.3	1.3	1.5	102.2	--

### Summary Per Cent Product Weight Loss

<u>Storage Environment</u>	<u>Type of Package</u>		
	<u>Marlex 6050 Laminate</u>	<u>Nylon 11 Laminate</u>	<u>Vitel 409 Laminate</u>
77° F, 50% RH	0.04	0.07	0.04
100° F, 10% RH	0.02	0.04	0.01
100° F, 90% RH	0.08	0.10	0.05

### Per Cent Oxygen in Head Space

77° F, 50% RH	5.7	4.9	4.5
100° F, 10% RH	7.4	5.8	5.9
100° F, 90% RH	11.0	6.2	10.7

A negligible sensory contribution from the three types of flexible packages was noted with the exception of the casein adhesive used to adhere the pouch to the protective jacket. (See Section 12.1, 12.2, and 12.4 for pack test details.)

We conclude that a flexible laminated pouch structure using 0.5 mil Mylar - 0.5 mil aluminum foil - 2 mil Nylon 11 with the pouch seal area supported in the protective jacket or by increasing the thickness of the Nylon 11 to 3 mil using the pouch center glued to the protective jacket will provide a satisfactory single service flexible package for irradiated chicken.

### 9.3 Single Service Flexible Package for Bacon

Of the three flexible packages extensively studied, the flexible laminate structure using Nylon 11 as the food contacting film is the recommended film for bacon.

The flexible pouch using Nylon 11 was the only structure to maintain adequate bond and seal strength under the test environments shown below:

#### Bond Strength\*

##### (Body Areas between Aluminum Foil and Food Contacting Film)

<u>Storage Environment</u>	<u>Type of Package</u>		
	<u>Marlex 6050 Laminate</u>	<u>Nylon 11 Laminate</u>	<u>Vitel 409 Laminate</u>
77° F, 50% RH	233	340	587
100° F, 10% RH	0	180	337
100° F, 90% RH	0	118	**

#### Bond Strength\*

##### (Seal Areas between Aluminum Foil and Food Contacting Film)

77° F, 50% RH	362	370	617
100° F, 10% RH	0	277	**
100° F, 90% RH	0	330	**

#### Seal Strength\*

77° F, 50% RH	3500	4633	2267
100° F, 10% RH	3533	4467	1433
100° F, 90% RH	3767	4533	950

\* grams/linear inch-width

\*\* Vitel 409 film too brittle to permit bond determination

### Head Space Gas Analysis in Can Control

	CO <sub>2</sub>	H <sub>2</sub>	O <sub>2</sub> + A	N <sub>2</sub>	CO	CH <sub>4</sub>	Total Head Space Volume	Vacuum In Can
77° F, 50 % RH	13.2	39.2	0.6	45.3	1.9	-	106.8	5.
100° F, 10% RH	15.4	32.6	0.7	49.7	1.8	-	88.5	3.3

### Summary Per Cent Product Weight Loss

<u>Storage Environment</u>	<u>Type of Package</u>		
	<u>Marlex 6050 Laminate</u>	<u>Nylon 11 Laminate</u>	<u>Vitel 409 Laminate</u>
77° F, 50% RH	0.07	0.08	0.06
100° F, 10% RH	0.04	0.08	0.03
100° F, 90% RH	0.08	0.13	0.06

### Per Cent Oxygen in Head Space

77° F, 50% RH	1.43	1.56	2.67
100° F, 10% RH	1.40	2.10	2.34
100° F, 90% RH	1.37	3.03	2.01

In comparison to the can control, a negligible sensory difference was detected in the bacon packaged in the three types of flexible packages. We conclude that the 0.5 mil Mylar - 0.5 mil aluminum foil - 2 mil Nylon 11 flexible laminate pouch with the pouch seal area supported in the protective jacket or by increasing the thickness of the Nylon 11 to 5 mil using the pouch center glued to the protective jacket will provide a satisfactory single service flexible package for irradiated bacon.

#### 10. RECOMMENDATIONS

We recommend that additional development be undertaken by the U. S. Army Natick Laboratories in their program for specifying flexible packages for pre-packed radiation sterilized foods for military usage:

a. Single service (4-5 oz.) flexible packages including protective jackets, using the 0.5 mil Mylar - 0.5 mil aluminum foil - 3 mil Nylon 11 laminate structure should be test packed with the radiation sterilized foods of greatest interest, e.g. ham, chicken, and bacon.

The test pack program should follow the general pattern described in this report.

b. Package integrity studies should be continued with additional correlation data obtained between a "laboratory" package integrity method, such as delineated in this report, the testing on the combat course, and actual field usage. One aspect of package integrity which should be evaluated but could not be included in this contract is single service package integrity as a part of a typical ration system.

c. A continuing package design program is warranted to provide systems for filling and sealing flexible packages for this use. Included in such a program should be materials and methods of protecting pouch seal areas. Any requirements for larger than 4 to 5 oz. single service packages will probably require different design concepts.

An additional design consideration is imposed by the use of electron beam radiation to sterilize the product. Consideration should be given to package handling and irradiation of pre-packaged pouches

which are jacketed after electron beam irradiation.

d. New, commercially available, food contacting polymeric films are frequently introduced to the flexible food packaging field. Those films which exhibit high resistance to fats, high bond and seal strength, and resistance to flexural fatigue should be screened. Polymeric films containing halogen groups should be very carefully screened for possible organoleptic contributions to the packaged food.

## 11. DEVELOPMENT AND FABRICATION OF FLEXIBLE POUCHES

### 11.1 Objective

To develop three types of flexible laminate pouches, utilizing commercially available materials and fabrication methods, as potential packages for radiation sterilized ham, chicken, and bacon. The three types of flexible pouches that were developed were combined as an integral part of protective paperboard jackets forming complete, 4 to 5 ounces, single service packages.

### 11.2 Program of Work

The flow diagram of work in this study is shown in Figure 6.

### 11.3 Screening Tests of Food Contacting Films

Based upon the results of the Phase I experiments, the preferred food contacting materials were polyamides, polyesters, and polyolefins. In Phase II, additional specific commercially available films of those polymer types were screened. In addition, a polycarbonate film was screened. Table 1 lists all of the films that were screened to obtain the three specific types to be intensively investigated.

Figure 6

DEVELOPMENT AND FABRICATION OF FLEXIBLE POUCHES

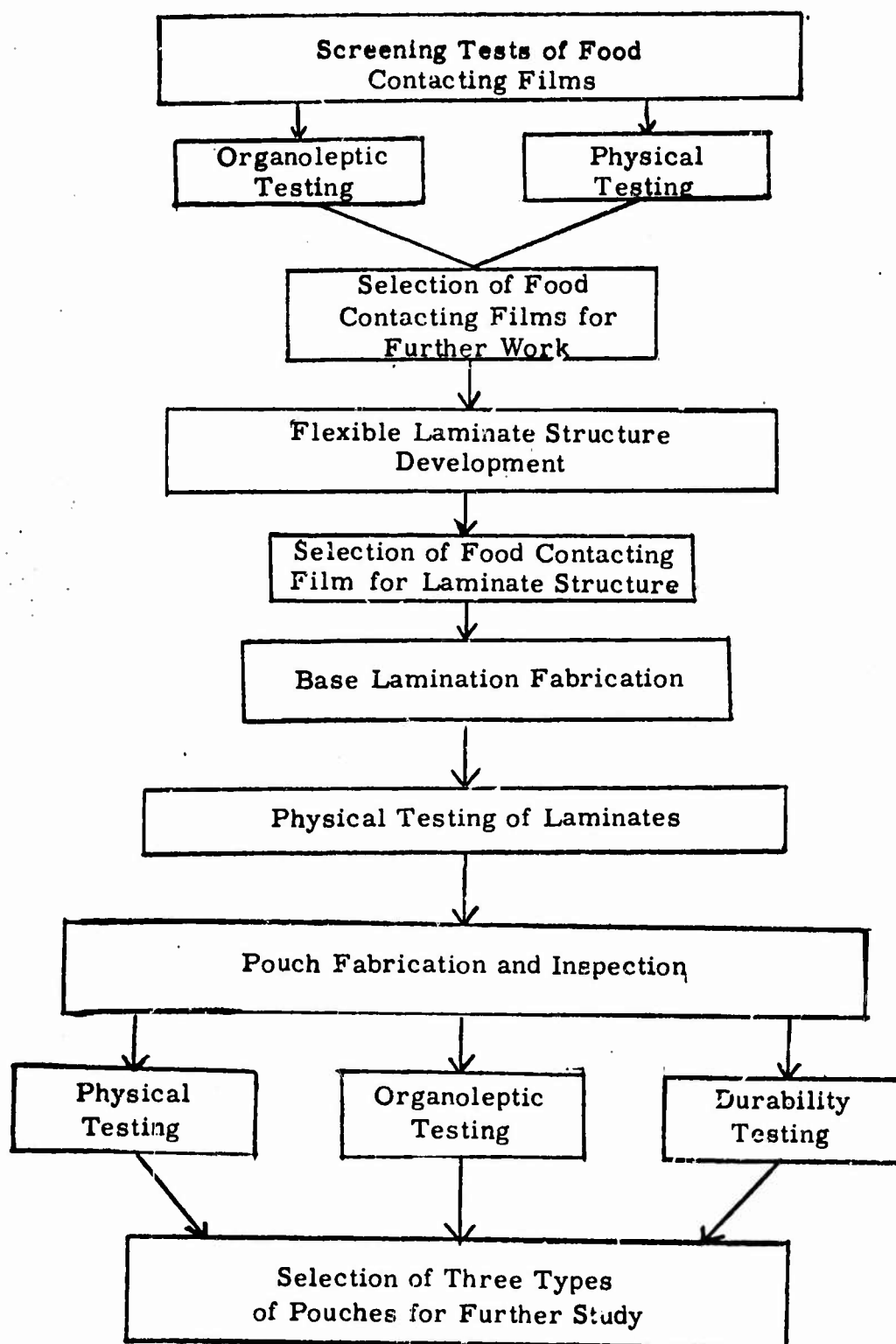


TABLE 1

SCREENING TESTS OF FOOD CONTACTING FILMS

List of Specific Films Tested

<u>Food Contacting Film</u>	<u>Trade Name</u>	<u>Supplier</u>
Polyvinylidene Chloride	Saran Wrap 18-L	Dow Chemical
Polyvinyl Chloride	VBA-3122	Cadillac Plastics
	VBA-9124	Cadillac Plastics
	F-11	Goodyear Tire & Rubber
	VBA-1142	Cadillac Plastics
	VBA-3027	Cadillac Plastics
Polyolefin	Petrothene 239	U. S. Industrial
	Polypropylene	Avisun Corp.
	Grex 2201-Polyethylene	W. P. Grace
	Polyallomer	Eastman Chemical
	Conaloy 2100 HY	Continental Can
Polysulfide	Marlex 6050	Phillips Petroleum
Polyamide	Rilsan (Nylon 11)	May Industries
	Nylon 6	Allied Chemical
Polyester	G-5	Minnesota Mining
	Mylar A	E. I. DuPont
	Kovar	Eastman Chemical
	Vitel 409	Goodyear Tire & Rubber
Polycarbonate	Lexan	General Electric
Polystyrene	Polyflex	Monsanto Chemical

### 11.31 Sample Preparation and Test Procedures

Twelve (12) sample pouches were prepared of each of the candidate materials (Table 1) in the 4-1/2" x 7" O.D. size. The pouches were sealed under optimum sealing conditions established by prior material testing. The fabricated pouches were then filled with 100 cc. of distilled water and the top seal closure made, after manually removing the air from the package, by sealing on a Sentinel impulse sealer. The prepared samples were then placed into paperboard jackets and separated into two groups. One group was irradiated to a dose of 6 megarads at the U. S. Army Natick Laboratories and the other held as the non-irradiated control. The samples after irradiation were then shipped to the Continental Can Company Technical Center for evaluation.

The sample pouches were evaluated organoleptically as follows: Pouches were cut open and the head space in the pouches sniffed by a seven-member panel (no more than two persons using the same pouch) and rated for odor. Ratings were given on a 1-to-100 scale, with 100 best and 70 considered to be the lowest level for acceptance. Irradiated and the non-irradiated control pouches were paired for comparison purposes. The results of the odor evaluations are tabulated in Table 2.

Subsequently, the distilled water was removed from the pouches and placed in clean 250 cc. beakers and numbered for identification. The irradiated and control samples were paired for comparison purposes. These samples were tasted by a seven-member panel and rated for flavor on a 1-to-100 scale, with 100 best and 70 considered to be the

lowest level for acceptance. An identified control sample of distilled water was provided for reference. The results of the taste evaluations are also tabulated in Table 2.

Seal strength was chosen as the preferred method of comparing the physical effects of irradiation on the single film pouches and, also, on the multiple ply laminate pouches evaluated in the screening experiments.

Two specimens for seal strength measurement were cut alternately from each side and the bottom of the pouch seal area for a total of six samples from each pouch. The emptied pouches used to obtain seal strength were the same pouches, irradiated and control, that were used in the organoleptic evaluation. Seal strengths obtained before and after irradiation are shown in Table 3.

#### 11.32 Summary of Results

The polystyrene films are satisfactory for organoleptic properties; however, the inherent brittleness of these films makes them unsuitable as an internal heat seal facing film for a shelf stable food product.

The polyvinylidene chloride films are unsuitable as an irradiated food contacting film because of off-odor and off-flavor development resulting from irradiation. The unplasticized polyvinyl chloride films, as illustrated by the VBA-3027 film, are borderline because of flavor development. However, in the plasticized form, the films are too brittle for use as a heat seal facing film for a shelf stable food

**TABLE 2**  
**FOOD CONTACTING MATERIALS SELECTION:**  
**Organoleptic Screening Tests - Acceptability (Water)**

Food Contacting Material*	Avg. Score (1)				Rating
	Irrad.		Control		
	T	O	T	O	
.001" Saran Wrap 18-L	69	56	91	71	U
.001" Vinyl VBA 3122	20	50	80	100	U
.003" Vinyl VBA 9124	20	60	80	100	U
.002" AT-3 Polypropylene	50		90		U
.002" Polyallomer	65		90		U
50M - 35F - .003" F-11 Vinyl	10	30	--		U
50M - 35F - .003" VBA 1142 Vinyl	20	30	--		U
.003" Petrathene 239	84-70	69	95	98	Q
.001" Petrathene 239	79-80	72	86	94	Q
.001" Vinyl VBA 3027	79		96		Q
50M - 35F - .003" Grex 2201	78		85		Q
.002" Polyallomer		70		80	Q
.001" Nylon 77C (2)	84	94	81	98	S
.001" Polyflex Polystyrene	83	87	98	99	S
.001" Mylar A Polyester	93	93	94	93	S
.0006" 6-5 Polyester	89	100	88	100	S
.0005" Kodar Polyester	88	100	88	100	S
.001" Lexan Polycarbonate	81	100	86	100	S
.0015" 2100HY Conaloy	79	80	90	80	S
.001" Al. Foil - .001" Vitel 409	82	100	87	100	S
50M - 35F - .001" Nylon 11	84	100	88	100	S
50M - 30F - .003" Marlex 6050	82		--		S
.001" Vinyl VBA 3027		90		97	S
.002" AT-3 Polypropylene		80		100	S
50M - 35F - .003" Grex 2201		90		90	S

T = Taste      O = Odor

(1) Rating on a 1-100 scale (100 best) 70 considered lowest level of acceptability. U = Unsatisfactory Q = Questionable S = Satisfactory

(2) Water developed yellow coloration in irradiation pouches

NOTE: The films were classified on the basis of a minimum score of 80 and at least 66% score above 70 being satisfactory film. An unsatisfactory film classification was a score of 70 or below and a questionable film would be an intermediate score between the satisfactory and unsatisfactory classifications.

\* In laminate structures the food contacting film is listed last.

TABLE 3

Screening Tests of Food Contacting Films Seal StrengthGrams/Lineal Inch Width<sup>(2)</sup>

<u>Types of Films and Laminates</u>	<u>Non-Irradiated</u>		<u>Irradiated</u>	
	<u>Avg.</u>	<u>Range</u>	<u>Avg.</u>	<u>Range</u>
.001" VBA 3122 Vinyl	1017	920-1100	995	800-1150
.003" VBA 9124 Vinyl	2967	2800-3100	3266	3100-3450
.0006" 6-5 Polyester	917	700-1080	700	440-1080
.0005" Kodar Polyester	733	330-1120	550	370- 720
.0005" Mylar A Polyester	518	320- 680	(1)	
.002" AT-3 Polypropylene	2583	2400-2650	2433	2360-2500
.001" Lexan Polycarbonate	2383	1950-2650	2000	1680-2340
.002" Polyallomer	2570	1800-3600	2890	2300-3600
.0015" 2100HY Conaloy	1600	1200-2000	2300	2200-2500
50M - 35F - .001" Nylon 11	3233	2550-3700	3070	2500-3300
50M - 35F - .003" F-11 Vinyl	3533	3350-3550	4133	3800-4450
50M - 35F - .003" VBA 1142 Vinyl	5483	5100-5950	2767	2500-3000
.001" Al. Foil - .001" Vitel 409	2617	1520-3450	1983	1200-2520
50M - 35F - .003" Grex 2201	4900	4700-5000	4430	4200-4700

(1) No Sample was available.

(2) Continental Can Test Method STM-13 ("Seal Strengths" (Appendix B)).

product. The plasticized films F-11, VBA-1142, VBA-3122, and VBA-9124 are unsatisfactory because of irradiation odor and flavor development. The polyolefin films have suitable seal strength factors but exhibit a wide range of organoleptic behavior. Polypropylene and polyallomer films were unsuitable because of off-flavor development on irradiation. The medium density Petrothene 239 was questionable on off-flavor and off-odor development; however, the higher density polyethylene Grex 2201 and Marlex 6050 were judged to be somewhat better. The Marlex 6050 was rated as the best polyolefin on organoleptic properties after irradiation. The Grex 2201 also performed well enough on organoleptic and seal properties to be considered for further evaluation.

The polyester films showed some seal strength loss due to irradiation but was not considered sufficient to cause problems in their usage in the laminated form. This group of films gave the best organoleptic performance both before and after irradiation. The G-5 Mylar and Kodar films do not heat seal readily; however, they can be sealed on thermal impulse equipment. Since the Mylar was deemed suitable and was also used for Phase I extraction work, it was evaluated in this phase as a polyester food contacting surface in the event the more experimental Vitel 409 did not function successfully. Since Vitel 409 provided good organoleptic and suitable heat seal properties, it was explored further for this usage. The polyamide films were judged to be suitable on organoleptic and seal properties. The Nylon 6 (77C) showed some yellow color development in the water samples after irradiation

which may be objectionable. The extraction data from Phase I indicated a much lower fat extraction with Nylon 6 than water extraction. Since high fat content meat products are to be packed, the Nylon 6 (77C) was included. The Nylon 11 provided very good seals and organoleptic performance.

The polycarbonate film showed very good organoleptic and seal properties.

The Conaloy (nylon-polyethylene alloy) showed good organoleptic and seal properties.

#### 11.4 Flexible Laminate Structure Development

The results of the screening tests (Section 11.3) indicated that the food contacting polymer types which should be further studied were high density polyethylene, Nylon 6, polycarbonate, Nylon 11, and polyester. Table 4 lists the specific polymers obtained for more extensive evaluation prior to the final selection of the three food contacting materials to be used in the remaining Phase II tests.

Table 4

#### FLEXIBLE LAMINATE STRUCTURE DEVELOPMENT

##### Selected Specific Food Contacting Materials

<u>Generic Type</u>	<u>Trade Name</u>	<u>Supplier</u>
Polyester	Vitel 409	Goodyear Tire & Rubber E. I. DuPont
	Mylar A	
Polyamide	Nylon 11	May Industries Allied Chemical
	Nylon 6	
Polyolefin	Grex 2201	W. R. Grace Phillips Petroleum Continental Can
	Marlex 6050	
	Conaloy 2100 HY	
Polycarbonate	Lexan	General Electric

#### 11.41 Fabrication of Laminates

Roll stock of the eight food contacting films were obtained from the suppliers (Table 4) and adhesive laminated to the foil side of the base lamination (9.31) using commercial scale equipment in a CCC Flexible Packaging Division plant. An attempt was made to extrusion laminate the Vitel 409 to the foil side of the base lamination. However, because of equipment, process and quality problems this approach was judged less desirable. Therefore, the Vitel 409 was purchased as a film and adhesive laminated to the foil side of the base lamination. All of the eight food contacting films were adhesive laminated to the foil side of the base lamination using L-4533, epoxy type adhesive. Sufficient lamination was prepared of each of the eight food contacting films to fulfill the requirements of the remaining packaging studies of the Phase II effort. The final laminations were aged for a period of one week prior to physical evaluation and pouch fabrication.

#### 11.42 Physical Testing of Laminates

Water vapor transmission rates were run on all laminates, flat and creased, by an industry standard method designated as Continental Can Company's test method STM-97. Samples, both before and after six megarads of irradiation in air, were checked for water vapor transmission rates. The results are presented in Table 5.

Since all of the laminate structures used 0.5 mil aluminum foil, the WVTR rates were extremely low and beyond the range of the standard test method to differentiate any difference; within the

laminate structures. In order to establish a differential in the test pouch structures of any magnitude, it would be necessary to run an extended testing period by this method, or use a long storage period pouch method. It was not felt that this would be significant in this program, since all of the materials have extremely good barrier properties, with no great significant difference being noted between the irradiated and non-irradiated units.

#### Internal Tear Resistance

Samples of all the laminate stocks, both before and after 6 megarads of irradiation, were checked for internal tearing using the Elmendorf Tear test unit following Continental Can Company Test Method STM-30 using a 2-1/2" sample and the results reported in terms of grams per 2-1/2" sample. The data is shown in Table 6.

The polyester and polycarbonate laminated films show the lowest internal tear resistance. Their tear resistance values do not seem to be noticeably affected by irradiation. However, the Grax 2201 did show a lowering on irradiation which was probably due to the irradiation effect on the rubber component. The Conaloy showed slight improvement in internal tear resistance, which may be due to some cross linking of the material. The Marlex 6050 and Nylon 11 laminates did not show any significant change on tear resistance due to irradiation. However, the Nylon 6 showed considerable lowering of tear strength. The results of this evaluation does show a preference for the polyolefin films for tear resistance.

### Tensile Strength

Tensile strength was measured on all laminate samples of the laminates both before and after 6 megarads of irradiation, using Continental Can Company's test method STM-57. The method utilizes a Scott tensile tester with a standard speed of 12 inches per minute with values reported in lbs./linear inch of width. (See Table 7 for the results.)

The tensile strength testing did not show any great difference between the irradiated and non-irradiated samples. However, as was expected, the Mylar laminate samples showed superior tensile strength to the other laminated samples, with polycarbonate laminate being the next best structure to the Conaloy laminate and the Grex laminate on the basis of tensile strength.

### Oxygen Permeability

Oxygen permeability rate was measured on all laminate samples both before and after 6 megarads of irradiation using Continental Can Company's test method STM-123, which utilizes American Instrument Company's air permeability apparatus. The values given are corrected for atmospheric pressure differentials and reported in terms of cc's per 100 sq. inches per 24 hours with 1 atmosphere differentials at 24°C. The data is shown in Table 8.

It is difficult to draw definite conclusions from the oxygen permeability data obtained from the laminated films. The fact remains that irradiation does not significantly affect the oxygen

permeability properties of the laminated test samples. However, it is felt that the pressure diffusion system of measuring oxygen permeability of films is not the best method of measuring permeability on foil laminates. A more practical pouch oxygen permeability method would be desirable. However, the length of time available for screening the laminates did not permit conducting pouch oxygen permeability on the samples. The oxygen permeability values could be misleading and should not be weighed heavily in the selection between the different materials.

#### Heat Seal Curves

In order to establish the seal properties of the laminate structures, heat seal curves were run only on the non-irradiated laminated samples using Continental Can Company test method STM-13, utilizing a Sentinel Model 12-AS heat sealer with one jaw heated using a 1/2 sec. dwell and 40 psi of pressure varying the temperature of 25°F increments. Heat seal curves were not run on irradiated samples since the seals would be made prior to the irradiation. The samples were cut on a Thwing Albert sample cutter to 1" width and the seal values pulled on an Alfred Suter tensile tester recording the values in grams per inch of width. Since materials are known to vary in the web and against the web direction, the heat seal curves were both in the web and against the web direction. Results of the heat seal curve studies are presented in Table 9.

The heat seal curves were run on roll stock within 3 days of laminating and it is felt that the heat seal values do not reflect the final expected heat seal strength of the laminates since

they probably had not been aged sufficiently from the time of lamination. It was necessary to run seal curves to establish the approximate heat seal temperatures necessary for use on the pouch fabricating equipment. The heat seal curves showed that it was impossible to effectively seal the Conaloy lamination by current heat or impulse sealing techniques. The Nylon 6 was found to be quite troublesome to heat sealing, particularly after moisture pickup resulting in blistering of the heat seal areas due to volatilization of the moisture. The heat seal curves of the Grex 2201 and the Marlex 6050 indicate some slightly linear response resulting in a slightly weaker seal in the cross web direction. The Nylon 11, polycarbonate and the Nylon 6 laminates require extremely high sealing temperatures when compared to the polyolefins and the Vitel 409 material (See Table 9).

#### Mullen Burst Resistance

Samples, both before and after irradiation in air at a dose of 6 megarads, were checked for Mullen Burst by Continental Can Company's test method STM-29, using a Perkins Model C Mullen tester, with the Mylar facing away from the diaphragm. The results are reported in lbs./sq. in. required to rupture the sample and are presented in Table 10.

No great difference could be noted between the non-irradiated and irradiated samples subjected to the Mullen burst test. The Mullen burst values generally tend to follow the tensile values. The Mylar laminate was far superior to the other materials, with the polycarbonate laminate next, and the Marlex 6050 laminate better than the other polyolefin laminates.

#### 11.43 Pouch Fabrication and Inspection

Since the heat seal curve table indicates that it is not practical to fabricate a heat sealed pouch from the laminate with the Mylar or Conaloy food contacting surfaces, these materials were not fabricated into pouches on production equipment. However, the other six materials were fabricated into pouches using a Model 21 Simplex Pouch Machine. Table 11 gives the top and bottom jaw temperatures, on the side and bottom seals, used to fabricate these pouches. The selection of these temperatures was guided by the seal curve, and the seal strength actually obtained on the fabricated pouches indicated on Table 12, which covers the seal testing of the pouches from the pouch machine using Continental Can STM-13. At the time of pouch fabrication the pouches were also tested by an Air Burst Test using Continental Can Test Method STM-147 (See Table 13). The pouch fabrication was adjusted to produce the highest attainable air burst value with 35 psi considered to be the lowest acceptable level. Inspection of the heat seal strengths was run in conjunction with the air burst test to assure that optimum heat seal strengths were achieved. A minimum of 4,000 grams/inch heat seal strength was the desired goal. Normal operational techniques and adjustments were required for pouch fabrication of these six food contacting film laminates. The heat seal strength variations on the Vitel and the slightly lower heat seal strengths on the polycarbonate were determined to be due to the lack of the complete cure (insufficient aging) of the laminate at the time of pouch fabrication.

**TABLE 5**  
**FLEXIBLE LAMINATE STRUCTURE DEVELOPMENT**

**WATER VAPOR TRANSMISSION RATES**

<u>Structure and Description</u>		<u>Gms/100 sq in/24 hrs @ 100°F, 95%RH (1)</u>			
		<u>Non-Irradiated</u>		<u>Irradiated</u>	
		<u>Flat</u>	<u>Creased</u>	<u>Flat</u>	<u>Creased</u>
50Mx50Fx.003" Grex 2201	Minimum	-.0021	-.0031	+.00068	+.00034
	Maximum	+.00085	-.0015	+.0014	+.0063
	Average	+.00063	-.0024	+.00098	+.0026
50Mx50Fx.003" Conaloy	Minimum	-.0022	-.00051	+.0010	0.0
	Maximum	-.00051	+.0026	+.0022	+.0032
	Average	-.0014	+.0015	+.0017	+.0017
50Mx50Fx.003" Marlex 6050	Minimum	-.00068	-.0014	+.0012	+.0017
	Maximum	+.0012	+.00017	+.0041	+.0041
	Average	+.0003	-.00058	+.0025	+.0028
50Mx50Fx.002" Nylon 11	Minimum	-.0024	-.00086	+.00068	+.00068
	Maximum	-.00068	+.0012	+.00086	+.0012
	Average	-.0013	-.00006	+.0008	+.00096
50Mx50Fx.002" Polycarbonate	Minimum	-.0014	-.0021	-.0075	+.00034
	Maximum	+.0019	+.0033	+.0014	+.0043
	Average	-.0001	-.00007	-.0031	+.0019
50Mx50Fx.002" Mylar A	Minimum	-.0011	-.0012	+.00051	+.0010
	Maximum	+.0026	+.0027	+.0021	+.0021
	Average	+.0003	+.0004	+.0014	+.0016
50Mx50Fx.002" Nylon 6	Minimum	-.00034	0.0	+.0086	+.0010
	Maximum	0.0	+.0041	+.0015	+.0015
	Average	-.00034	+.0021	+.0012	+.0012
50M x 50Fx.002" Vitel 409	Minimum	-.0084	-.00034	+.00068	+.00034
	Maximum	+.0039	+.00068	+.0109	+.0065
	Average	-.0011	+.00006	+.0058	+.0031

(1) Continental Can Company Test Method - STM 97 (Appendix F)

TABLE 6

FLEXIBLE LAMINATE STRUCTURE DEVELOPMENT

## INTERNAL TEAR RESISTANCE

Structure and Description		Values given in gms/2-1/2" sample (1)			
		<u>Non-Irradiated</u>		<u>Irradiated</u>	
		M.D.	C.D.	M.D.	C.D.
50Mx50Fx.003"					
Grex 2201	Minimum	128	176	112	128
	Maximum	176	256	128	160
	Average	149	219	117	149
50Mx50Fx.003"					
Conaloy	Minimum	112	112	112	128
	Maximum	128	160	144	160
	Average	117	139	128	149
50Mx50Fx.003"					
Marlex 6050	Minimum	112	192	128	176
	Maximum	112	208	160	208
	Average	112	197	139	187
50Mx50Fx.002"					
Nylon 11	Minimum	96	112	96	128
	Maximum	112	112	112	144
	Average	107	112	107	133
50Mx50Fx.002"					
Polycarbonate	Minimum	64	64	64	64
	Maximum	80	80	80	80
	Average	69	75	75	75
50Mx50Fx.002"					
Mylar A	Minimum	80	96	80	96
	Maximum	96	112	96	96
	Average	91	101	91	96
50Mx50Fx.002"					
Nylon 6	Minimum	128	128	96	96
	Maximum	144	144	96	112
	Average	133	139	96	101
50Mx50Fx.002"					
Vitel-409 Polyester	Minimum	48	64	48	64
	Maximum	64	80	64	80
	Average	53	75	58.1	69.3

(1) Continental Can Company Test Method - STM 30 (Appendix D)

TABLE 7

FLEXIBLE LAMINATE STRUCTURE DEVELOPMENT

TENSILE STRENGTH

<u>Structure &amp; Description</u>		<u>Lbs./Linear In-Width (1)</u>			
		<u>Non-Irradiated</u>		<u>Irradiated</u>	
		<u>M.D.</u>	<u>C.D.</u>	<u>M.D.</u>	<u>C.D.</u>
50M-50F-.003" Grex 2201	Minimum	19.5	21.8	19.5	21.5
	Maximum	19.8	21.9	20.1	21.8
	Average	19.7	21.8	19.8	21.7
50M-50F-.003" Conaloy	Minimum	22.0	21.9	24.0	21.2
	Maximum	24.3	23.1	24.9	22.8
	Average	22.9	22.6	24.4	22.0
50M-50F-.003" Marlex 6050	Minimum	24.2	23.4	24.9	23.4
	Maximum	26.1	23.7	27.4	24.6
	Average	25.0	23.5	25.8	23.8
50M-50F-.002" Nylon 11	Minimum	24.0	22.5	24.5	22.9
	Maximum	24.1	23.0	25.4	23.7
	Average	24.1	22.7	25.0	23.4
50M-50F-.002" Polycarbonate	Minimum	29.3	29.1	30.1	29.6
	Maximum	32.2	29.8	32.3	30.4
	Average	30.7	29.4	31.0	30.0
50M-50F-.002" Mylar A	Minimum	43.0	70.0	53.0	65.0
	Maximum	58.5	75.0	60.0	73.0
	Average	49.5	72.7	56.0	69.3
50M-50F-.002" Nylon 6	Minimum	22.2	24.7	24.2	24.6
	Maximum	25.4	24.8	28.2	26.9
	Average	24.1	24.7	26.3	25.4
50M-50F-.002" Vitel 409 Polyester	Minimum	20.6	19.4	26.0	22.0
	Maximum	22.5	20.6	28.5	22.5
	Average	21.3	19.9	26.9	22.2

(1) Continental Can Company Test Method - STM 57 (Appendix E)

TABLE 8

FLEXIBLE LAMINATE STRUCTURE DEVELOPMENTOXYGEN PERMEABILITY

		cc/100 sq. in./24 hrs. @ 1 Atmosphere Differential 24°C (1)			
<u>Structure &amp; Description</u>		<u>Non-Irradiated</u>		<u>Irradiated</u>	
		<u>Flat</u>	<u>Creased</u>	<u>Flat</u>	<u>Creased</u>
50M-50F-.003" Grex 2201	Minimum	.3	.2	.7	.8
	Maximum	.6	.9	1.1	1.1
	Average	.46	.56	.8	1.0
50M-50F-.003" Conaloy	Minimum	.8	1.1	.7	1.3
	Maximum	1.1	3.5	1.5	2.3
	Average	1.0	2.3	1.1	1.7
50M-50F-.003" Marlex 6050	Minimum	1.0	1.3	1.5	2.7
	Maximum	1.3	2.0	2.3	4.3
	Average	1.2	1.8	1.9	3.5
50M-50F-.002" Nylon 11	Minimum	.6	.6	1.0	.3
	Maximum	2.0	4.3	1.9	4.6
	Average	1.4	3.0	1.6	2.3
50M-50F-.002" Polycarbonate	Minimum	1.0	4.8	2.65	4.6
	Maximum	2.1	5.3	5.64	6.6
	Average	1.5	5.0	3.8	5.6
50M-50F-.002" Mylar A	Minimum	1.2	6.5	2.3	3.9
	Maximum	3.4	7.2	6.8	6.7
	Average	2.3	6.9	3.8	5.4
50M-50F-.002" Nylon 6	Minimum	1.1	1.3	1.0	1.2
	Maximum	2.3	5.0	4.9	4.8
	Average	1.7	2.9	2.4	3.1
50M-50F-.002" Vitel 409 Polyester	Minimum	.5	.8	.9	2.0
	Maximum	1.0	3.2	2.5	4.2
	Average	.7	1.7	1.9	3.1

(1) Continental Can Company Test Method STM 123 (Appendix G)

TABLE 9

## FLEXIBLE LAMINATE STRUCTURE DEVELOPMENT

## HEAT SEAL CURVES

Structure and Description	Sentinental Heat Sealer 1 Jaw Heated-1/2 Sec. Dwell 40 PSI Suter Grams/Inch									
	225°F.	350°F.	375°F.	400°F.	425°F.	450°F.	475°F.	500°F.	525°F.	550°F.
50M-50F-.003" M.D. Grex 2201	N/S	N/S	3833F (3800- 3850)	4200 (4150- 4250)	4416 (4400- 4450)	4316 (4250- 4400)	4500 (4450- 4550)	4333 (4300- 4400)	--	--
	N/S	2950 (2850- 3000)	3166F (3100- 3300)	3616 (3600- 3650)	3483 (3450- 3500)	3916 (3900- 3950)	3716 (3600- 3800)	3483 (3450- 3500)	--	--
50M-50F-.003" M.D. Marlex 6050	N/S	N/S	2250 (2100- 2400)	5083F (4850- 5300)	5733 (5700- 5750)	5683 (5650- 5700)	5816 (5700- 6000)	5816 (5750- 5900)	--	--
	N/S	N/S	866 (800- 980)	4316F (4200- 4500)	4900 (4700- 4950)	5050 (4800- 5200)	5083 (5050- 5100)	5133 (4950- 5350)	--	--
50M-50F-.002" M.D. Nylon 11	N/S	N/S	N/S	N/S	N/S	2620 (2250- 3000)	4166F (4100- 4300)	5050 (5000- 5150)	--	--
	N/S	N/S	N/S	N/S	N/S	3450 (3300- 3700)	3583F (3500- 3650)	4583 (4500- 4700)	--	--
50M-50F-.002" M.D. Polycarbonate	N/S	N/S	N/S	N/S	N/S	400 (250- 500)	2866 (2750- 3000)	3500F (3500- 3500)	--	--
	N/S	N/S	N/S	N/S	N/S	516 (450- 550)	1583 (1450- 1800)	3416F (3350- 3500)	--	--

TABLE 9

## FLEXIBLE LAMINATE STRUCTURE DEVELOPMENT

## HEAT SEAL CURVES - (Continued)

Structure and Description	Sentinel Heat Sealer 1 Jaw Heated-1/2 Sec. Dwell 40 PSI Super Grams/Inch									
	325°F.	350°F.	375°F.	400°F.	425°F.	450°F.	475°F.	500°F.	525°F.	550°F.
50M-50F-.002" M.D., Nylon 6				333 (250- 450)	1100 ( 800- 1300)	2433F (2300- 2600)				
C.D.				503 (430- 600)	733 (600- 800)	2866F (2750- 3000)				
50M-50F-.002" M.D. Vitel 409 Polyester	216 (200- 240)	1826 (1780- 1900)	3516F (3450- 3600)	3533 (3400- 3600)	3550 (3450- 3600)	3616 (3500- 3700)	3450 (3300- 3550)			
C.D.	766 (660- 840)	713 (620- 980)	2916F (2800- 3000)	3616 (3500- 3750)	3100 (3000- 3200)	3483 (3250- 3700)	3500 (3250- 3700)			
50M-50F-.003" M.D. Conafloy	N/S	N/S	N/S	N/S	N/S	N/S	N/S	162 (150- 180)	233D (220- 230)	410D (370- 440)
C.D.	N/S	N/S	N/S	N/S	N/S	N/S	N/S	160 (150 170)	203D (200 210)	346D (320- 360)

50M-50F-.002" The stock was not heat sealable but could be impulse sealed and seal conditions established on impulse of 2 seconds with 2 second cooling and 20 psi gauge super grams/inch of M.D. 3983 (3850-4100) C.D. 6300 (5900-6700).

F - Fusion Point M.D. - Machine Direction  
D - Distortion C.D. - Cross Direction N/S No Seal

TABLE 10

FLEXIBLE LAMINATE STRUCTURE DEVELOPMENTMULLEN BURST RESISTANCE

		<u>Lbs./Sq. In. (1)</u>	
<u>Structure &amp; Description</u>		<u>Non-Irradiated</u>	<u>Irradiated</u>
50M-50F-.003" Grex 220	Minimum	52	48
	Maximum	55	53
	Average	53.2	50.8
50M-50F-.003" Conaloy	Minimum	57	58
	Maximum	67	65
	Average	63.0	61.3
50M-50F-.003" Marlex 6050	Minimum	58	62
	Maximum	66	71
	Average	63.3	66.7
50M-50F-.002" Nylon 11	Minimum	57	58
	Maximum	60	60
	Average	58.5	59.3
50M-50F-.002" Polycarbonate	Minimum	70	68
	Maximum	79	74
	Average	74.3	70.0
50M-50F-.002" Mylar A	Minimum	157	150
	Maximum	163	159
	Average	159	153.5
50M-50F-.002" Nylon 6	Minimum	58	58
	Maximum	66	52
	Average	60.8	59.9
50M-50F-.002" Vitel 409 Polyester	Minimum	46	52
	Maximum	56	60
	Average	50.3	56.7

(1) Continental Can Company Test Method STM-29 (Appendix C)

TABLE 11

FLEXIBLE LAMINATE POUCH DEVELOPMENT

SEAL CONDITIONS FOR POUCH FABRICATION

<u>Structure</u>	<u>Top Jaw</u>	<u>Bottom Jaw</u>	<u>Bottom Seal</u>
50M-50F-.003" Grex 2201	445	445	480
50M-50F-.003" Marlex 6050	460	460	500
50M-50F-.002" Nylon 11	555	550	565
50M-50F-.002" Polycarbonate	520	515	485
50M-50F-.002" Nylon 6	560	555	565
50M-50F-.002" Vitel 409 Polyester	480	480	500

MODEL #21 Simplex Pouch Machine

Temperature of Sealer Jaw Surfaces in °F

**TABLE 12**

**FLEXIBLE LAMINATE POUCH DEVELOPMENT**

**FABRICATED POUCH SEAL STRENGTH**

<u>Structure</u>	<u>Gms./Linear Inch Width (1)</u>			
	<u>Side Seal</u>		<u>Bottom Seal</u>	
	<u>Average</u>	<u>Range</u>	<u>Average</u>	<u>Range</u>
50M-50F-.003" Grex 2201	5000	4500-5500	4500	4500-4600
50M-50F-.003" Marlex 6050	5730	5300-6000	5030	4800-5400
50M-50F-.002" Nylon 11	5300	5000-5600	5100	4600-5300
50M-50F-.002" Polycarbonate	3960	3700-4200	4280	4200-4500
50M-50F-.002" Nylon 6	5600	5400-5800	5400	5200-5600
50M-50I-.002" Vitel 409 Polyester	4410	3900-4850	4190	3100-4900

(1) Continental Can Company Test Method STM-13 (Appendix B)

TABLE 13

FLEXIBLE LAMINATE POUCH DEVELOPMENT

AIR BURST TEST

<u>Structure Description</u>	<u>Pouch Rupture/p.s.i. (1)</u>	
	<u>Average</u>	<u>Range</u>
50M-50F-.003" Grex 2201	41	39-45
50M-50F-.003" Marlex 6050	39	38-40
50M-50F-.002" Nylon 11	40	40-40
50M-50F-.002" Polycarbonate	38	38-39
50M-50F-.002" Nylon 6 (Capran 77C)	35	35-35
50M-50F-.002" Vitel 409 Polyester	38	38-38

(1) Continental Can Company Test Method STM-147 (Appendix H)

All test pouches satisfied the minimum air burst requirements. To maintain a project time schedule, all of the pouches were fabricated in spite of substandard seal strength in some cases. The high pouch heat seal strength values do not agree with the low air burst values obtained on Nylon 6. This inconsistency appears to be due to the moisture in the Nylon causing blistering in the heat seal area. The blisters undoubtedly contributed to the weak air burst results.

The pouch fabrication results showed that the Grex 2201, Marlex 6050, Nylon 11, polycarbonate and Vitel 409 laminates could be fabricated into pouches with excess of 4,000 grams seal on standard production pouch equipment. However, the Grex 2201 showed a much weaker bottom seal than did the Marlex 6050, apparently due to the linearity of the former film. It was not possible to produce as high a seal strength on the Nylon 6, apparently due to the moisture effect on the film. All of these pouches gave the desired 35 psi minimum pouch burst failure with the Nylon 6 at the lower level of this specification (See Table 13).

#### 11.5 Comparative Evaluation of Six Types of Flexible Pouches

To provide a basis for selecting three of the six types of flexible pouches, which survived the preliminary screening tests, an additional series of physical and organoleptic evaluations were performed.

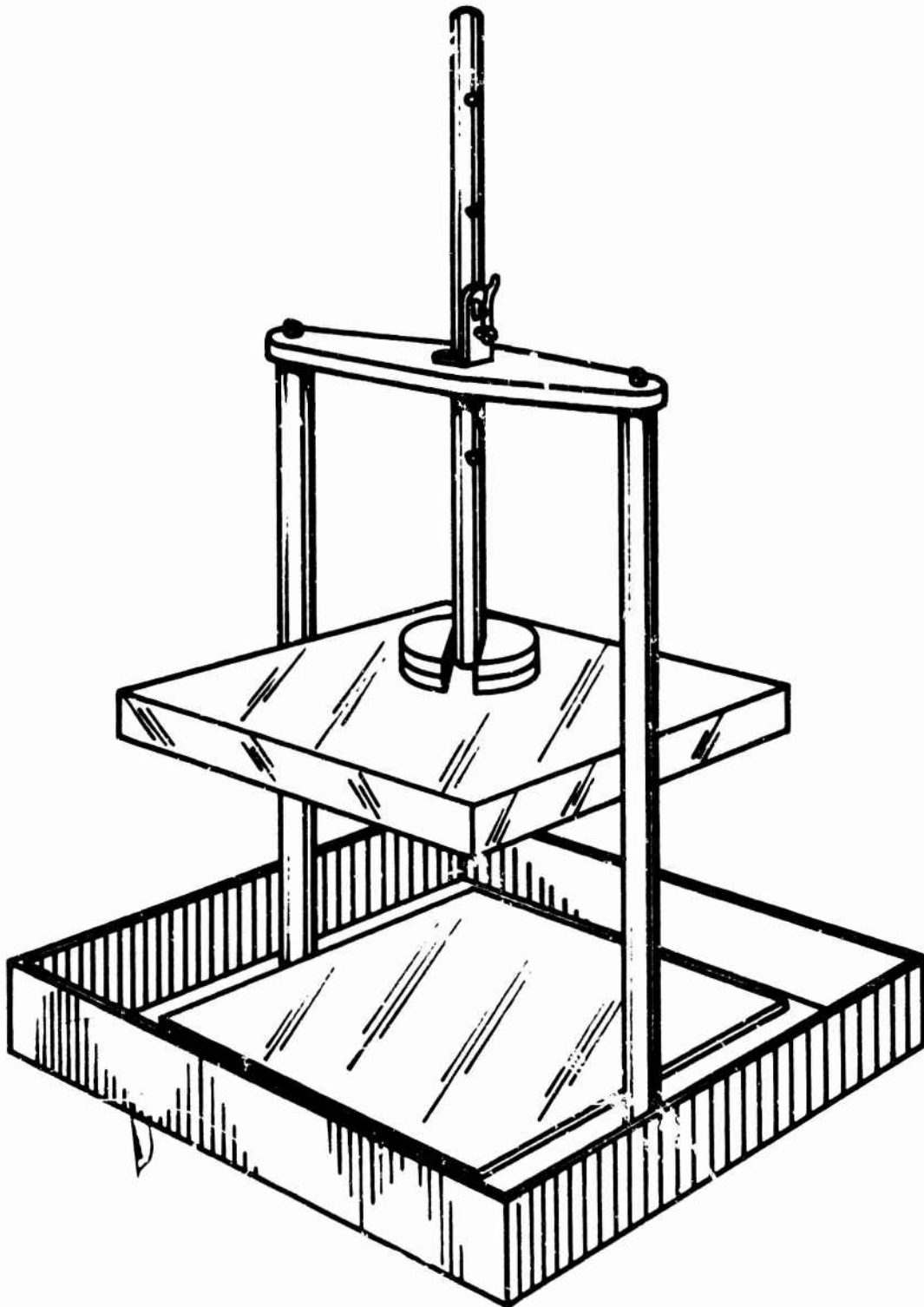
##### 11.51 Physical Evaluation

Comparative durability of the six types of flexible pouches was determined as follows:

### Impact Fatigue

Pouches of the six pouch types were filled with 5 oz. of water, heat sealed, and tested using method CCC STM-152A (See Appendix I). This method consists of placing theunjacketed pouch on a platform and dropping a weighted plate from increasing heights until rupture occurs. The test is started by dropping the smallest weight at the lowest height a minimum of three times. If the pouch passes, the drop height is increased to a maximum of 13-1/2", then additional weight is added for dropping at the minimum height and increasing up to 13-1/2". When failure is encountered a minimum of six pouches are tested at that weight and drop height. The total number of drops required for failure of the six pouches are recorded and averaged. The results of that test are shown as follows:

FIGURE 7



DROP TESTER

# IMPACT FATIGUE TEST

<u>Structure Description</u>	<u>3.132 lb. Weight</u> <u>Impact Height</u>			<u>8.6 lb. Weight</u> <u>Impact Height</u>	
	<u>3"</u>	<u>6"</u>	<u>13-1/2"</u>	<u>3"</u>	<u>6"</u>
50M-50F-.003" Grex 2201	P	P	P	7(1-17)	0
50M-50F-.003" Marlex 6050	P	P	P	2(1-3)	0
50M-50F-.002" Nylon 11	P	P	P	4(1-10)	0
50M-50F-.002" Polycarbonate	P	P	P	P	1.8(1-3)
50M-50F-.002" Mylar A **	9 (2-14)	0	0	0	0
50M-50F-.002" Nylon 6	P	P	2.3 (2.3)	0	0
50M-50F-.002" Vitel 409 Polyester	P	2.6 (2.3)			

P = Passed

0 = Complete Failure

\*\* Hand made pouches - not possible to make on production equipment.

### Shipping Impact Test

Forty eight each of the six types of pouches were filled with 5 oz. of water, heat sealed, enclosed in protective jackets, and positioned in a corrugated inner shipping carton. The cartons were dropped on the base end onto a metal plate from a height of three feet. The pouches were inspected for failure after every five drops, until a total of twenty drops were completed. The results are reported below:

#### SHIPPING IMPACT TEST

<u>Structure Description</u>	<u>% Failure/3 ft. ht.</u>		
	<u>5 drops</u>	<u>10 drops</u>	<u>20 drops</u>
50M-50F-.003" Grex 2201	0	0	0
50M-50F-.003" Marlex 6050	0	0	0
50M-50F-.002" Nylon 11	0	0	0
50M-50F-.002" Polycarbonate	0	0	0
50M-50F-.002" Nylon 6	100(1)	-	-
50M-50F-.002" Vitel 409 Polyester	0	0	0

### Vibration Testing

Forty eight of each type of pouch were filled with 5 oz. of water, containing a dye(2), placed in protective jackets without gluing (to exaggerate abuse) were exposed to the vibrating table using test method ASTM-D999-63T (1 g. of force). All pouches were removed from vibrating unit at the end of 1/2 hour, opened and checked for leakage.

- (1) All failures were top seal failures. Blistering occurred in the seal areas due to moisture pick-up. Desiccating the pouches immediately prior to sealing would improve sealing.
- (2) Refer to MIL-B-131B, 4.5.2.3.2 Aerosol O. T. 1% or equivalent (Methylene blue dye added).

If no leakage occurred, they were placed back on the vibrating table for an additional hour and again removed and inspected. The test was then discontinued. The results of this evaluation are reported below:

#### VIBRATION TEST

<u>Structure Description</u>	<u>% Failure (4)</u>	
	<u>1/2 Hour Exposure</u>	<u>1 Hour Exposure</u>
50M-50F-.003" Grex 2201	0	0
50M-50F-.003" Marlex 6050	0	0
50M-50F-.002" Nylon 11	0	0
50M-50F-.002" Polycarbonate	0	44 <sup>(2)</sup>
50M-50F-.002" Nylon 6	31 <sup>(1)</sup>	44 <sup>(1)</sup>
50M-50F-.002" Vitel 409 Polyester	0	12.5 <sup>(3)</sup>

(1) All failures were top seal failures due to heat sealing problems.

(2) Failures were pin hole or flex fatigue failures along edge of seal or in body of pouch.

(3) Failures were flex fatigue failures in body or pouch.

(4) ASTM-D999-63T, Procedure A1G.

#### 11.52 Organoleptic Screening

As test media, distilled water and ground ham were selected for the following reasons:

a. Organoleptic differences are readily observed in distilled water.

b. Ground ham, one of the three types of foods which was test packed, was considered to be a more severe test medium than solid ham since it provides a greater surface area to volume ratio.

Twenty-four empty pouches each of the six food contacting film laminates were submitted to the U. S. Army Natick Laboratories along with thermally processed cans of ground ham. One set of the pouches was filled with ground ham and the other set was filled with distilled water. The filled and sealed pouches were exposed to a dosage of 6 megarads and returned to Continental Can Company, Chicago, for organoleptic evaluation.

The ham samples were rated for odor and color by a seven member panel on a 1-to-100 scale (100 best), with 70 considered the lowest level for acceptability. The results are shown below:

#### ACCEPTABILITY (GROUND HAM)

<u>Structure Description</u>	<u>Odor</u>		<u>Color</u>	
	<u>Average Score(1)</u>	<u>% Score Above 70</u>	<u>Average Score(1)</u>	<u>% Score Above 70</u>
50M-50F-.003" Grex 2201	61	17	55	0
50M-50F-.003" Marlex 6050	80	33	73	33
50M-50F-.002" Nylon 11	88	100	81	67
50M-50F-.002" Polycarbonate	91	100	91	100
50M-50F-.002" Nylon 6	87	83	78	67
50M-50F-.002" Vitel 409 Polyester	90	83	90	100

(1) Score was based on a 1-100 scale (100 best) and 70 considered lowest acceptable level as a gross judgment test.

A twelve man panel rated the odor difference between the irradiated ground ham in a metal can ("C" inside enamel) compared to the product irradiated in the flexible pouches as follows:

GROUND HAM IRRADIATED IN POUCHES VS. GROUND HAM IN METAL CANS

<u>Structure Description</u>	<u>Irradiation Odor Difference</u>
50M-50F-.003" Grex 2201	Significant (Higher)
50M-50F-.003" Marlex 6050	Not Significant
50M-50F-.002" Nylon 11	Not Significant
50M-50F-.002" Polycarbonate	Significant (Higher)
50M-50F-.002" Nylon 6	Not Significant
50M-50F-.002" Vitel 409 Polyester	Significant (Higher)

The results of taste testing the irradiated distilled water from the six types of packages are shown below:

ORGANOLEPTIC SCREENING TESTS-ACCEPTABILITY (WATER)

<u>Structure Description</u>	<u>Average Score (1)</u>	<u>% Score Above 70</u>
50M-50F-.003" Grex 2201	66	40
50M-50F-.003" Marlex 6050	82	80
50M-50F-.002" Nylon 11	84	80
50M-50F-.002" Polycarbonate	82	60
50M-50F-.002" Nylon 6	70(2)	40
50M-50F-.002" Vitel 409 Polyester	86	80

(1) Score was based on a 1-100 scale (100 best) and 70 considered lowest acceptable level. Triple distilled water control considered to be 100 on this scale.

(2) Water turned yellow after irradiation.

A twelve men panel rated the taste difference between distilled water irradiated in a metal can ("C" inside enamel) and in the six types of pouches as follows:

ORGANOLEPTIC SCREENING TESTS - DIFFERENCE

(WATER IRRADIATED IN POUCHES VS. WATER IN METAL CANS)

<u>Structure Description</u>	<u>Difference</u>
50M-50F-.003" Grex 2201	Highly Significant
50M-50F-.003" Marlex 6050	Significant
50M-50F-.002" Nylon 11	Highly Significant
50M-50F-.002" Polycarbonate	Significant
50M-50F-.002" Nylon 6(1)	Significant
50M-50F-.002" Vitel 409 Polyester	Significant
(1) Water had yellow tint.	

11.6 Selection of Three Types of Pouches for Intensive Studies

In cooperation with the Project Officer, U. S. Army Natick Laboratories, the results of the flexible package development were reviewed and the following three types of pouches were selected for the remaining tests in Phase II:

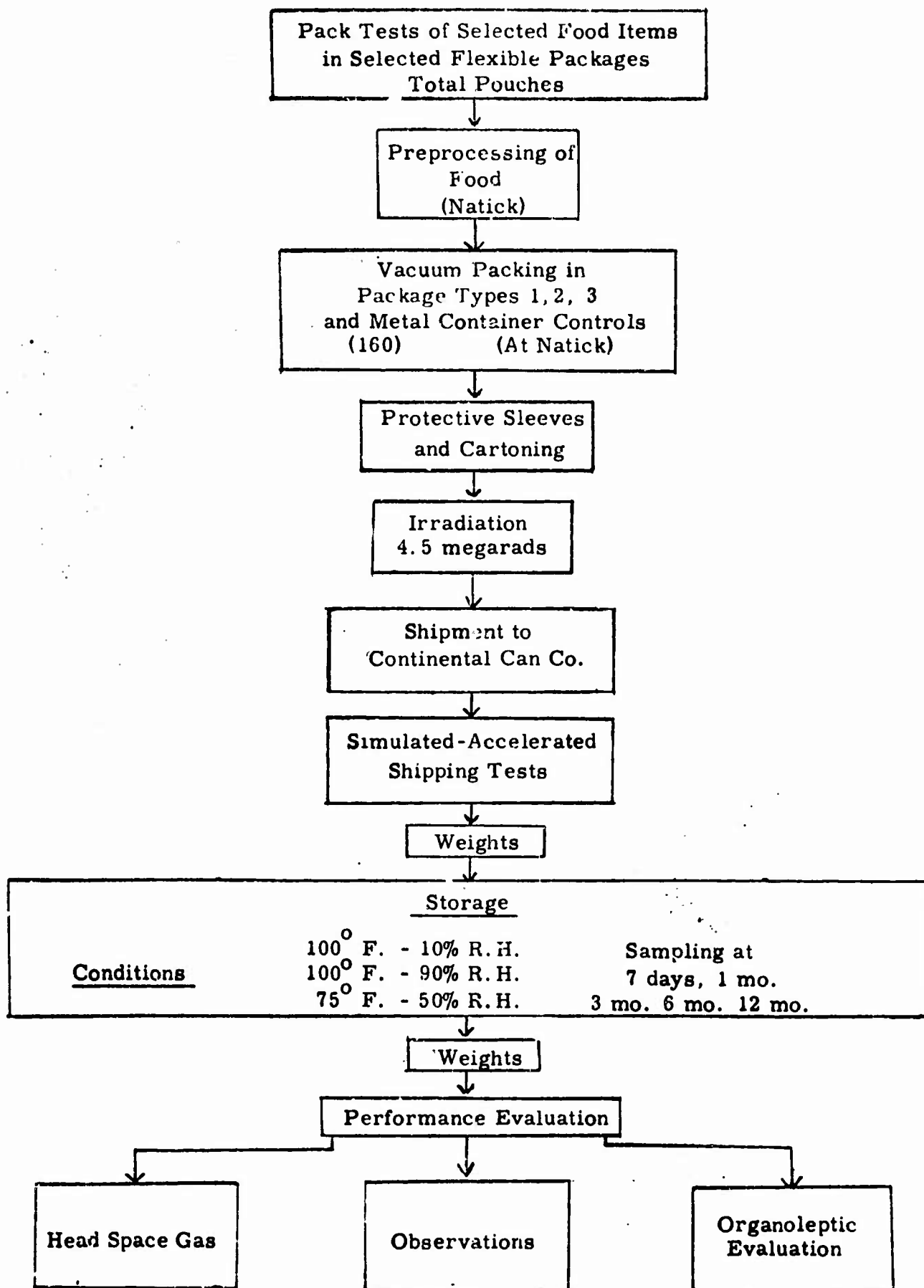
50M-50F-.003" Marlex 6050

50M-50F-.002" Nylon 11

50M-50F-.002" Vitel 409

The 50M-50F-.002" Grex pouch was rejected on the probable unacceptable organoleptic performance.

The 50M-50F-.002" Polycarbonate pouch was not selected because of severe flexural fatigue failures.



The 50M-50F-.002" Nylon 6 pouch developed a pronounced yellow discoloration when in contact with water and showed poor durability since it was difficult to heat seal without blistering. Hence, it was not selected for further testing.

## 12. PACK TEST OF HAM, CHICKEN, AND BACON

### 12.1 Objective

To determine the packaging performance of the three selected flexible packages at three test environments packed with radiation sterilized ham, chicken, and bacon. (See Figure 8.)

### 12.2 Methods

Based upon results of the pouch development program (See Section 11) the following three food contacting film laminated pouches were selected for the food irradiation pack test:

- (1) 50M-50F-.003" Marlex 6050
- (2) 50M-50F-.002" Nylon 11
- (3) 50M-50F-.002" Vitel 409

Five hundred (500) pouches fabricated from each of the three laminate structures, along with the required protective jackets, inner corrugated cartons and outer corrugated shippers (See Section 8.5) were forwarded to the U. S. Army Natick Laboratories for the pack test.

Sample pouches of each of the three selected laminates were sent to the U. S. Army Natick Laboratories for top sealing on their Flex-Vac 6-9 machine at the recommended seal temperatures (established from

TABLE 10

IRRADIATED POUCH TEST PACK:

TOP SEAL STRENGTH WITH FLEX-VAC 6-9 MACHINE

<u>Pouch Structure</u>	<u>Seal Temp. °F.</u>	<u>Grams/inch-width (1)</u>		
		<u>Pouch #1</u>	<u>Pouch #2</u>	<u>Pouch #3</u>
50M-50F	400-425	3800 F	3650 F	4350 F
.003" Marlex 6050	400-425	4100 F	3500 F	4000 F
(Seal edge very irregular due to heat creep.)				
50M-50F	475-500	3500 F	3000 F	3100 F
.002" Nylon 11	475-500	1900 F	2550 F	3200 F
(Seal edge very irregular due to heat creep.)				
50M-50F	475-500	3700 F	3450 F	3600 F
.002" Vitel 409	475-500	3450 F	3500 F	3450 F
(Seal edge very irregular due to heat creep.)				

NOTE: F indicates material fused at seal junction with seal failing at seal edge.

(1) Continental Can Company Test Method STM-13 (Appendix B)

heat seal curves; see Table 11). These sealed pouches were then returned to the Continental Can Company Technical Center and checked for heat seal strength. The results of the pouch seal tests are reported in Table 14. Since these results indicated undesirable seal characteristics, it was recommended that the Flex-Vac 6-9 be checked by the U. S. Army Natick Laboratories to improve seal characteristics. This was completed with some improvement in seal; however, in order to assure maximum seal for the pack test, a Sentinel Model 24A heat sealer was used to overseal the Flex-Vac 6-9 top seal.

An experimental food pre-processing and packaging line was set up at the U. S. Army Natick Laboratories. Personnel from that laboratory and from the Continental Can Company Technical Center performed the packing and processing operations during the week of October 26-31, 1964.

#### Handling of Selected Foods

The three selected meat products, ham, chicken, and bacon, were supplied by the U. S. Army Natick Laboratories for this pack test.

#### Ham

Pre-processed ham rolls were stored at 38°F prior to slicing and cutting of pieces approximately 4" x 3" x 1/2" in size. Approximately 4 ounces of meat was weighed and packed in a flexible pouch. Non-sliced ham rolls were packed in 603 x 700 metal cans for use as comparative controls.

#### Chicken

Pre-processed chicken breast rolls were stored at 38°F prior

to slicing and cutting of pieces approximately 4" x 2-7/8" x 1/2" in size. Approximately 4 ounces of meat was weighed and packed in a flexible pouch. Non-sliced chicken breast rolls were packed in 401 x 411 metal cans for use as comparative controls.

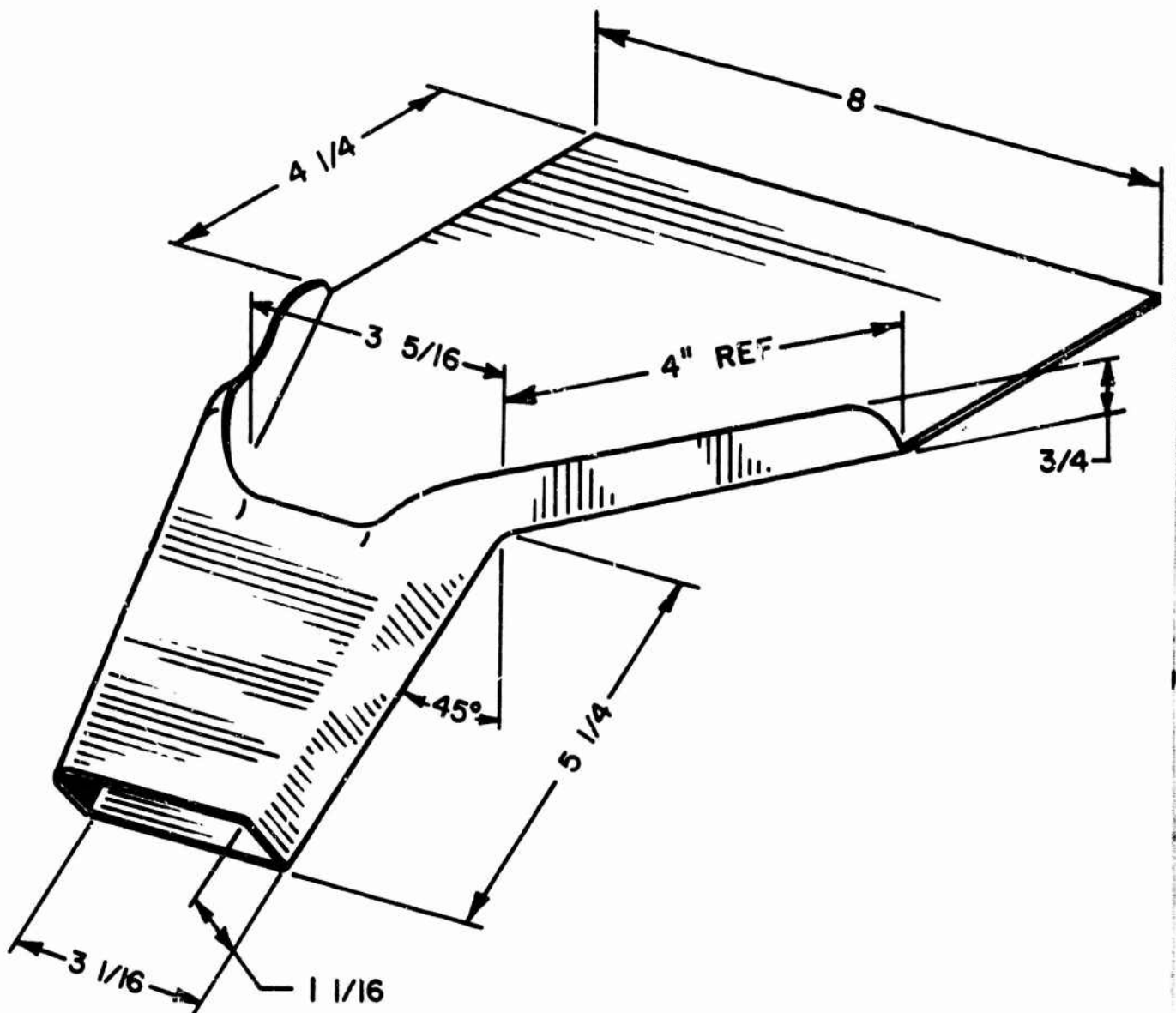
#### Bacon

Sliced, uncooked bacon was stored at 38°F prior to cutting of pieces approximately 4" x 1" x 6/64" in size. A total of 4 ounces of meat was packed in a flexible pouch. Shingles of sliced bacon were placed on parchment paper and packed in 307 x 509 metal cans for use as comparative controls.

#### Filling of Pouches

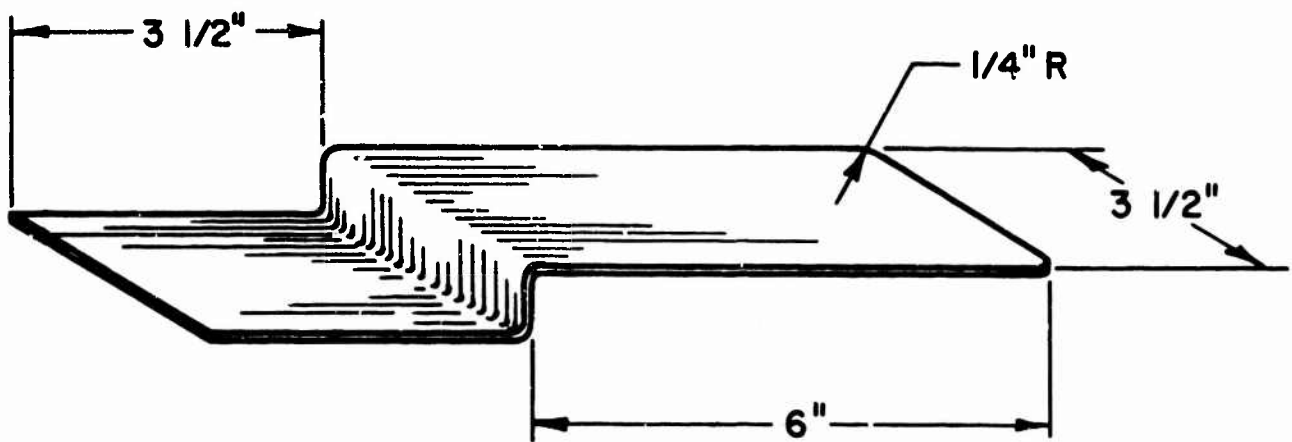
A pouch was opened and inserted onto the loading funnel (Figure 9). The prepared, sliced and weighed meat unit was placed on the loading spatula (Figure 10) and inserted into the loading funnel. The product was then pushed through the funnel. The filled pouches were placed upright in a tray for conveying to the vacuum sealing operation. The loading funnel was attached to the edge of a stainless working table at a convenient location in the packaging line between the product trimming and vacuum sealing operations. It was found necessary to wipe the loading funnel exterior surface, in the area where the pouch is inserted, after every 25 uses with a clean paper towel to remove product to prevent seal area contamination. This was more of a problem in packing chicken or ham.

FIGURE 9



LOADING FUNNEL

FIGURE 10



LOADING SPATULA

#### Vacuum Sealing

The filled pouches were immediately inserted into the Flex-Vac 6-9 unit and vacuum sealed with care exercised to obtain a flat, wrinkle free, top seal closure. The unit was adjusted for the vacuum to be in excess of 28" when checked in a vacuum bell jar. Immediately after sealing on the Flex-Vac 6-9, a full 1" width seal was imposed over the Flex-Vac seal by means of the Sentinel Model 24A heat sealer to assure a positive top seal. The sealing temperatures used on the pouch units were:

	<u>Flex-Vac 6-9</u>	<u>Sentinel Model 24A</u>
50M-50F-.003" Marlex 6050	500°F	500°F
50M-50F-.002" Nylon 11	550°F	500°F
50M-50F-.002" Vitel 409	500°F	500°F

A pressure of 40 psi and 1/2 sec. jaw dwell time was used as the other sealing conditions for both heat sealers.

#### Assembly in Protective Jacket and Shipping Containers

The protective jacket units were then coated with the National Starch Resin #36-6262 on the inside of the jacket, using a small size paint brush to coat approximately one-third of the pouch contact area. These pouches were then immediately placed in the protective jackets and inserted into the inner corrugated cartons and placed in 38°F storage for holding prior to radiation. All pouches, jackets and inner corrugated cartons were marked with a marking pen for identification.

### Irradiation Procedure

Two inner corrugated cartons, containing 48 individual pouches per carton, were removed from the 38°F storage area for each irradiation period of approximately one hour. The minimum dose received in any portion of the carton was 4.5 megarads; the maximum dose in any portion of the carton was 4.5 megarad plus 12%. Ferric-copper dosimetry, using one carton with an array of dosimetry ampules, was performed during this series of irradiation.\* Following irradiation, all inner corrugated cartons were inserted in outer corrugated shippers for shipment to the Continental Can Company Technical Center.

### 12.3 Package Evaluation Procedures

The radiation processed food packages, on arrival at the Continental Can Company Technical Center, were all subjected to a vibration test for 30 minutes in accordance with method ASTM-D999-63T. All food packages were then identified by marking the jackets with the code ledger including type of laminate, food product, storage conditions, inspection period and food package number. All of the pouches and blanks (sealed empty pouches in jackets) were weighed and placed in either the medium (50% RH, 77°F), low (10% RH, 100°F), or high (90% RH, 100°F) storage condition. At each inspection period (7 days, 3 months, 6 months, and 12 months), the food packages were removed from test areas, conditioned at 77°F, 50% RH for 24 hours, and weighed to note weight change. The food pouches were inspected for delamination, checked for head space oxygen and observable effects.

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\* All dosimetry was performed under the direction of R. D. Jarrett, Sr., U. S. Army Natick Laboratories, Natick, Massachusetts.

The examination samples were then transferred to the Food Sensory and Microbiology Sections, Metal Division R & D, for odor analysis and extraction of samples for toxin testing.

Pouches were opened aseptically; the product was handled as shown in Appendix O. After a portion of food had been removed the pouches were resealed using an impulse sealer, the resealed pouches and the control cans were stored at 38-40°F pending results of the mouse toxicity tests. Since the mouse tests were negative, the food was removed and prepared for physical and organoleptic testing.

#### Physical Testing

After removal of the food from the pouch, which was accomplished by removing the top seal, the emptied test pouches were then inspected for seal strength on side and bottom seal and bond of food contacting film to the aluminum foil in the body and seal areas.

#### Organoleptic Testing

Testing procedures were used to establish any potential organoleptic effect of the packaging materials on the quality of the foods as part of the evaluation of package performance of the three selected flexible pouches under study. Since the overall objective of this contract is the development of suitable flexible laminate packages for the three selected foods, the emphasis of the organoleptic evaluation was directed towards possible differences in the organoleptic suitability of the three package types as compared to the same foods that have been processed by irradiation in the identical manner but packaged in rigid

metal cans with an inside "C" enamel. The rigid metal container, previously approved as an "acceptable" package, was used as a control for measuring the unknown performance of the flexible laminate package materials.

Determinations of sensory difference were conducted with the Multiple Sample Difference Test, a modified paired comparison method.

"Flavor difference" scoring was numerically rated as follows:

- 0 - no difference
- 1 - very slight difference
- 2 - slight difference
- 3 - moderate difference
- 4 - large difference
- 5 - extremely large difference

The term "difference" was used to express a comparison with the food in the metal can controls.

The detailed procedures for the organoleptic evaluation are presented in Appendix F "Outline for Flavor Testing of Irradiated Meat," of this contract.

#### 12.4 Ham Pouch Test Pack

##### Ham Specification

The ham processed by the U. S. Army Natick Laboratories for this pack test was commercial, Grade A, fully cooked, smoked, rolled ham (in casing with metal ends), cut in slices 9/16" and trimmed on two sides to make a 3" x 4-1/2" steak.

#### 12.41 Physical Tests (1 week, 3, 6, and 12 months storage)

##### Weight Check

All pouch samples were checked, weighed at the end of one week, 3 months, 6 months, and 12 months as described under Section 10.27. The results of those weight checks are reported in Table 15 and show an average less than 0.1% weight change on all samples. Since this weight loss is less than 0.1% at the more severe desiccating exposure of 10% RH, it is concluded that this weight loss is insignificant on these packages at the 12 months' storage period and this weight change is too low to cause any serious problems with these products.

##### Head Space Gas

The ham test packages were analyzed for head space oxygen using Continental Can Test Method STM-179 (See Appendix J). All packages from this inspection period were checked by this method. Because of lack of contingency samples, total gas volume was only checked initially. However, it was observed on the packages that the volume of gas appeared visibly to remain fairly constant through the 12 months' storage period. The gas volume in the pouches was checked initially by using Continental Can Test Method STM-180 (See Appendix K). The total volume of head space in the can controls was evaluated by a double expansion technique and the head space gas was analyzed by standard chromatography techniques. The results of the can head space gas analysis are reported in Table 16 and show that the vacuum in the cans remain

TABLE 15

PACK TEST - HAM

Product Weight Loss

<u>Structure - Storage</u>	<u>Percent Product Weight Loss</u>			
	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>	
	<u>Average</u>	<u>Average</u>	<u>Average</u>	<u>Range</u>
<u>77°F, 50% R.H.</u>				
50M-50F-.003" Marlex 6050	<0.1	<0.1	0.1	0.1-0.12
50M-50F-.002" Nylon 11	<0.1	<0.1	0.09	0.07-0.1
50M-50F-.002" Vitel 409	<0.1	<0.1	0.07	0.05-0.08
<u>100°F, 10% R.H.</u>				
50M-50F-.003" Marlex 6050	<0.1	<0.1	0.1	0.09-0.11
50M-50F-.002" Nylon 11	<0.1	<0.1	0.1	0.09-0.11
50M-50F-.002" Vitel 409	<0.1	<0.1	0.05	0.04-0.06
<u>100°F, 90% R.H.</u>				
50M-50F-.003" Marlex 6050	<0.1	<0.1	0.1	0.07-0.20
50M-50F-.002" Nylon 11	<0.1	<0.1	0.1	0.06-0.1
50M-50F-.002" Vitel 409	<0.1	<0.1	0.1	0.07-0.12

fairly constant through the 12 months' storage. The oxygen and argon contents are reported together, but the analysis indicates that the oxygen accounted for 50% of the initial composition and that the oxygen content had dropped to 0% after the one week inspection period and was essentially 0% at the 3, 6, and 12 months' storage period in the can controls.

The oxygen content in the head space on the ham pouches is reported in Table 17 and shows a general increase in oxygen content on all the test samples from the 1 week through the 12 months' storage. After the 12 months' storage period, oxygen content appears to be approximately the same on all test samples. This level of oxygen content is normal for flexible package materials and should not greatly hamper the quality level of the contained products, although it is higher than the can control. The higher range of the 50M-50F-.003" Marlex 6050 at 100°F 10% RH is probably due to lowering of the foil-marlex bond at the 12 months' storage period.

#### Bond Strengths

The bond strengths were checked in the seal areas and the body areas between the foil and food contacting films using Continental Can Test Method STM-8 (See Appendix A). The results of the bond determinations were reported in Table 18 through 20. Due to a handling error, the empty ham packages from the one-week storage at 50% RH, 77°F and 10% RH, 100°F were disposed of after sampling of the ham, thus bond strength data could not be obtained on these packages. Heat sealing results show an initial increase in bond strength in the seal

**IRRADIATED POUCH TEST PACK**

### Head Space Gas Analysis - (Canned Ham Controls)

[illegible]

130°F., 10% R.H.

Vacuum Inches	-	-	11	11
Head Space (ml)	658.5	782.8	640.9	661.2
%CO <sub>2</sub>	21.4	19.0	18.8	20.0
%H <sub>2</sub>	21.5	22.3	23.5	23.4
%O <sub>2</sub> /A	1.2	1.4	0.7	0.7
%N <sub>2</sub>	53.0	54.0	53.6	53.0
%CO	2.3	2.6	2.8	2.3
%CH <sub>4</sub>	0.6	0.7	0.6	0.6
Total Per Cent	100.0	100.0	100.0	100.0

TABLE 17

IRRADIATED POUCH TEST PACK

Head Space Oxygen - (Ham Pouches)

<u>Storage and Structure</u>	<u>Head Space (ml)</u>		<u>1 Week</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
<u>77°F., 50% R. H.</u>						
50M-50F-.003 Marlex 6050	11	Avg. Range	0.56 0.29-1.01	0.99 0.94-1.04	1.19 1.01-1.47	1.27 0.97-1.86
50M-50F-.002 Nylon 11	10	Avg. Range	0.52 0.32-0.83	1.04 0.89-1.18	1.12 1.06-1.22	1.23 1.12-1.37
50M-50F.002 Vitel 409	11	Avg. Range	0.37 0.27-0.57	1.01 0.94-1.05	0.97 0.93-1.04	1.30 1.12-1.47
<u>100°F., 10% R.H.</u>						
50M-50F-.003 Marlex 6050	11	Avg. Range	0.70 0.33-1.22	0.84 0.68-0.93	1.02 0.94-1.12	2.67 1.27-5.17
50M-50F-.002 Nylon 11	10	Avg. Range	0.62 0.38-1.08	0.99 0.94-1.04	0.94 0.85-1.04	1.15 1.05-1.04
50M-50F-.002 Vitel 409	11	Avg. Range	0.49 0.38-0.60	0.87 0.76-1.03	1.00 0.96-1.06	1.20 1.10-1.30
<u>100°F., 90% R.H.</u>						
50M-50F-.003 Marlex 6050	11	Avg. Range	0.58 0.38-0.90	0.72 0.49-0.85	0.93 0.63-1.22	1.13 1.04-1.19
50M-50F-.002 Nylon 11	10	Avg. Range	0.77 0.39-1.39	1.10 1.03-1.15	0.96 0.85-1.04	1.13 1.05-1.20
50M-50F-.002 Vitel 409	11	Avg. Range	0.50 0.27-0.87	0.97 0.90-1.14	1.01 0.95-1.12	1.35 1.13-1.80

area. However, at the 3 months' inspection period, the bond strength in the seal area decreases and is essentially equivalent to the bond strength in the body area at the 3, 6, and 12 months' inspection period.

The bond strengths decreased from the initial bond values on all samples. These values were stable at the 3 and 6 months' levels, however, marked changes were noted at the 12 months' level. The 50M-50F-.002" Nylon 11 bond strengths were maintained sufficiently high under all test conditions to be functional through the 12 months' storage period. The 50M-50F-.003" Marlex 6050 maintained functional bond level under storage at 50% RH, 77°F. However, it suffered serious bond loss at the 100°F, 10% RH, apparently due to the fat permeating the polyolefin film at these elevated temperatures. While this is undesirable at the 12 months' storage, it should be pointed out that the bond strengths were maintained at the elevated temperature conditions at the 6 months' level with this Marlex 6050 laminate. This factor should be taken into consideration in reviewing the failure at the 12 months' level.

The 50M-50F-.002" Vitel 409 material showed satisfactory bond maintenance through the 6 months' storage period. However, at the 12 months' storage period, the film became too brittle to permit bond determinations, due to degradation of the film. This was evident at 50% RH, 77°F, as well as the 100°F temperature and variable humidity conditions. This would indicate there was a stabilization problem with the Vitel 409 film at this 12 months' storage period in contact with the fat from the ham food product.

### Seal Strengths

The seal strengths were checked on the side seals and bottom seals (in order to include the two directions of the stock) by using Continental Can's Test Method STM-13 (See Appendix B). The results of the seal strength testing are reported in Tables 21 through 26. Due to handling error, the empty ham pouches from the 1 week storage from 50% RH, 77°F and 10% RH, 100°F were disposed after sampling the product, thus seal strength data could not be obtained on these samples. The 50M-50F-.002" Nylon 11 seal strengths decreased slightly from the initial values at the 1 week period and remained fairly constant throughout the 12 months' storage period at all test conditions indicating good stability and adequate functional seal strengths maintained through the 12 months' storage period. The 50M-50F-.003" Marlex 6050 showed bond lowering from the initial value at the 1 week period, with side seals being maintained satisfactorily throughout the 12 months' storage period under all test conditions. Some variation was noted at the various inspection levels on the side seals; however, this was felt to be due more to variation in samples, rather than the product effect. The bottom seals were lowered considerably at the 1 week period with a gradual general lowering up to the 6 months' period, then maintained at this level through the 12 months' storage period. Though the bottom seals initially were almost as strong as the side seals, the bottom seals after the 12 months' storage were only 50% of the side seal values under all three test conditions. This is probably due to the linear polymeric structure

TABLE 18

IRRADIATED POUCH TEST PACK

Body Area Bond Strengths - (77°F, 50% R.H. - Ham Pouch)

<u>Structure - Body Area (1)</u>		<u>Grams/Linear Inch Width (3)</u>			
		<u>Initial (2)</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
<u>50M-50F-.003"</u> <u>Marlex 6050</u>	Average	910			
	Range	850-950			
Right Side Area	Average		380	490	360
	Range		350-450	350-550	350-400
Left Side Area	Average		370	470	350
	Range		320-500	350-550	300-380
Bottom Area	Average		360	490	360
	Range		350-400	350-600	350-400
<u>50M-50F-.002"</u> <u>Nylon 11</u>	Average	560			
	Range	560-560			
Right Side Area	Average		310	400	340
	Range		280-350	350-450	300-350
Left Side Area	Average		300	380	360
	Range		230-450	230-450	300-400
Bottom Area	Average		370	450	350
	Range		320-450	450-450	300-400
<u>50M-50F-.002"</u> <u>Vitel 409</u>	Average	593			
	Range	510-650			
Right Side Area	Average		420	410	(4)
	Range		320-450	350-450	
Left Side Area	Average		440	460	(4)
	Range		350-500	430-520	
Bottom Area	Average		410	410	(4)
	Range		300-480	350-450	

- (1) Body areas between the foil and food contacting film.  
 (2) Bonds checked on stock at the time pouches were manufactured.  
 (3) Continental Can Company Test Method STM-8 (Appendix A).  
 (4) Vitel 409 film too brittle to permit bond determination.

TABLE 19

IRRADIATED POUCH TEST PACKBody Area Bond Strengths - (100°F, 10% R.H. - Ham Pouch)

<u>Structure - Body Area (1)</u>		<u>Grams/Linear Inch Width (3)</u>			
		<u>Initial (2)</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
<u>50M-50F-.003"</u> <u>Marlex 6050</u>	Average Range	910 850-950			
Right Side Area	Average Range		350 250-450	340 200-450	130 0-300
Left Side Area	Average Range		350 300-450	370 250-450	180 0-250
Bottom Area	Average Range		370 300-450	340 300-350	70 0-280
<u>50M-50F-.002"</u> <u>Nylon 11</u>	Average Range	560 560-560			
Right Side Area	Average Range		350 300-420	350 280-450	260 250-300
Left Side Area	Average Range		320 300-270	370 340-450	270 150-300
Bottom Area	Average Range		300 280-330	390 350-450	280 250-300
<u>50M-50F-.002"</u> <u>Vitel 409</u>	Average Range	593 510-650			
Right Side Area	Average Range		440 350-500	510 400-700	(4)
Left Side Area	Average Range		430 400-500	540 450-650	(4)
Bottom Area	Average Range		400 380-430	560 500-700	(4)

(1) Body areas between the foil and food contacting film.

(2) Bonds checked on stock at the time pouches were manufactured.

(3) Continental Can Company Test Method STM-8 (Appendix A).

(4) Vitel 409 film too brittle to permit bond determination.

TABLE 20

IRRADIATED POUCH TEST PACK

Body Area Bond Strengths - (100° F., 90% R.H. - Ham Pouches)

Structure-Body Area (1)	Initial (2)	Grams/Linear Inch Width (3)			
		1 Week	3 Months	6 Months	12 Months
50M-50F-.003' Marlex 6050	910 850-950				
Right Side Area	Avg. Range	390 300-470	370 330-460	510 500-550	0 0 - 0
Left Side Area	Avg. Range	480 300-730	400 320-640	480 350-550	0 0 - 0
Bottom Area	Avg. Range	360 320-470	410 330-560	470 400-550	0 0 - 0
50M-50F-.002' Nylon 11	560 560-560				
Right Side Area	Avg. Range	530 400-580	430 280-530	430 350-500	250 200-300
Left Side Area	Avg. Range	460 250-520	430 320-600	400 300-500	290 250-300
Bottom Area	Avg. Range	350 280-450	350 310-450	340 300-380	300 300-300
50M-50F-.002' Vitel 409	593 510-650				
Right Side Area	Avg. Range	580 500-730	520 430-680	490 400-600	(4)
Left Side Area	Avg. Range	670 440-380	490 250-630	500 400-650	(4)
Bottom Area	Avg. Range	550 350-650	470 340-560	550 450-720	(4)

- (1) Body areas between the foil and food contacting film.  
 (2) Bonds checked on stock at the time pouches were manufactured.  
 (3) Continental Can Company Test Method STM-8 (Appendix A).  
 (4) Vitel 409 film too brittle to permit bond determination.

of the Marlex 6050 film and the effect of the high fat content food product weakening of the film in the web direction on these sample pouches. The 50M-50F-.002" Vitel 409 structure showed a gradual seal strength lowering at each inspection period in the test program at the elevated temperature and variable humidity conditions, with these strengths becoming quite low at the 6 and 12 months' inspection period at the 100°F, 90% R.H. and 100°F, 10% R.H. exposure. However, at the 77°F, 50% R.H. exposure, the seal strengths at the 12 months' period, though lower than initial, were still of a sufficiently high enough range to be functional. This Vitel 409 laminate would not be suitable for storing this high fat product at the elevated temperature conditions due to the seal loss. However, it may be suitable from a seal standpoint at standard room conditions. This seal strength lowering at the elevated temperatures appears to be due to the fat effect on the film and this factor should be explored further in regards to the Vitel 409 film.

#### Observations

Even though the bond strength fell to zero (0) on the 50M-50F-.003" Marlex 6050 samples, there was no ply separation on any of the test samples. The 50M-50F-.002" Vitel 409 film became extremely brittle after the 12 months' storage period and resulted in inability to measure bond and lowering of seal strength on the laminate. However, this did not result in observable seal breaks or seal failure on this structure. There were no other package changes observed on these samples, other than the above reported bond and seal variations.

TABLE 21

IRRADIATED POUCH TEST PACKSeal Area Bond Strength - (77°F, 50% R.H. - Ham Pouches)

<u>Structure - Seal Area (3)</u>		<u>Grams/Linear Inch Width (1)</u>		
		<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
<u>50M-50F-.003" Mariex 6050</u>				
Right Seal Area	Average	450	450	420
	Range	400-480	350-500	400-500
Left Seal Area	Average	460	430	410
	Range	400-550	300-520	350-450
Bottom Seal Area	Average	450	430	400
	Range	450-470	300-550	350-450
<u>50M-50F-.002" Nylon 11</u>				
Right Seal Area	Average	500	370	350
	Range	450-520	280-400	350-350
Left Seal Area	Average	450	320	380
	Range	340-560	300-400	350-450
Bottom Seal Area	Average	420	410	400
	Range	270-500	400-450	400-400
<u>50M-50F-.002" Vitel 409</u>				
Right Seal Area	Average	520	370	(2)
	Range	430-600	350-400	
Left Seal Area	Average	510	420	(2)
	Range	400-600	400-500	
Bottom Seal Area	Average	490	360	(2)
	Range	400-550	300-400	

(1) Continental Can Company Test Method STM-8 (Appendix A).

(2) Vitel 409 film too brittle to permit bond determination.

(3) Seal areas between the foil and food contacting film.

TABLE 22

IRRADIATED POUCH TEST PACKSeal Area Bond Strength - (100°F, 10% R.H. - Ham Pouches)

<u>Structure - Seal Area (3)</u>		<u>Grams/Linear Inch Width (1)</u>		
		<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
<u>50M-50F-.003" Marlex 6050</u>				
Right Seal Area	Average	400	270	210
	Range	(350-480)	(200-300)	( 0-450)
Left Seal Area	Average	420	320	240
	Range	(380-520)	(200-400)	( 0-450)
Bottom Seal Area	Average	440	310	250
	Range	(400-500)	(300-350)	( 0-450)
<u>50M-50F-.002" Nylon 11</u>				
Right Seal Area	Average	460	310	325
	Range	(400-500)	(250-400)	(300-350)
Left Seal Area	Average	420	320	300
	Range	(350-480)	(300-400)	(200-350)
Bottom Seal Area	Average	360	340	310
	Range	(340-400)	(300-400)	(300-350)
<u>50M-50F-.002" Vitel 409</u>				
Right Seal Area	Average	500	480	(2)
	Range	(400-550)	(380-640)	
Left Seal Area	Average	490	480	(2)
	Range	(450-550)	(370-620)	
Bottom Seal Area	Average	470	440	(2)
	Range	(450-500)	(320-550)	

- (1) Continental Can Company Test Method STM-8 (Appendix A).  
 (2) Vitel 409 film too brittle to permit bond determination.  
 (3) Seal areas between the foil and food contacting film.

TABLE 23

IRRADIATED POUCH TEST PACKSeal Area Bond Strength - (100°F, 90% R.H. - Ham Pouches)

<u>Structure - Seal Area (3)</u>		<u>Grams/Linear Inch Width (1)</u>			
		<u>1 Week</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
<u>50M-50F-.003" Marlex 6050</u>					
Right Seal Area	Average	810	490	390	
	Range	(740-860)	(430-530)	(350-480)	
Left Seal Area	Average	840	470	360	
	Range	(780-930)	(340-650)	(300-380)	
Bottom Seal Area	Average	740	490	370	
	Range	(500-920)	(400-660)	(350-400)	
<u>50M-50F-.002" Nylon 11</u>					
Right Seal Area	Average	900	530	360	300
	Range	(800-1000+)	(450-580)	(280-450)	(250-350)
Left Seal Area	Average	780	530	350	300
	Range	(560-1000+)	(450-690)	(300-500)	(250-350)
Bottom Seal Area	Average	930	520	290	325
	Range	(860-1000+)	(480-550)	(250-320)	(300-350)
<u>50M-50F-.002" Vitel 409</u>					
Right Seal Area	Average	790	610	500	(2)
	Range	(700-870)	(510-650)	(400-600)	
Left Seal Area	Average	790	600	430	(2)
	Range	(650-880)	(410-780)	(400-500)	
Bottom Seal Area	Average	720	560	510	(2)
	Range	(550-870)	(440-620)	(400-700)	

- (1) Continental Can Company Test Method STM-8 (Appendix A).  
 (2) Vitel 409 film too brittle to permit bond determination.  
 (3) Seal areas between the foil and food contacting film.

TABLE 24

IRRADIATED POUCH TEST PACKSeal Strengths - (77°F, 50% R.H. - Ham Pouches)

<u>Structure - Seal Area</u>		<u>Grams/Linear Inch Width (1)</u>			
		<u>Initial (2)</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
<u>50M-50F-.003" Marlex 6050</u>					
Right Seal	Average	5700	5200	4100	4800
	Range	5300-6000+	4600-5700	2200-5000	2600-5400
Left Seal	Average	5700	5500	5100	4800
	Range	5300-6000+	5000-5800	4500-5600	4200-5200
Bottom Seal	Average	5000	3200	3100	3300
	Range	4800-5400	1500-4500	1600-4500	2600-4700
<u>50M-50F-.002" Nylon 11</u>					
Right Seal	Average	5300	4600	4700	4800
	Range	5000-5600	4100-5000	4400-5200	4400-5100
Left Seal	Average	5300	4200	4500	4800
	Range	5000-5600	3800-4800	3800-4900	4700-5000
Bottom Seal	Average	5100	3800	4300	4400
	Range	4600-5300	2500-4900	2000-5000	3400-5100
<u>50M-50F-.002" Vitel 409</u>					
Right Seal	Average	4400	4400	3700	2900
	Range	3900-4900	4200-4600	1300-4400	2200-3500
Left Seal	Average	4400	4500	3900	4300
	Range	3900-4900	4200-5000	3600-4300	4000-4500
Bottom Seal	Average	4200	4400	4000	4000
	Range	3100-4900	3900-4800	3400-4800	3700-4200

(1) Continental Can Company Test Method STM-13 (Appendix B).

(2) Seals checked on stock at the time pouches were manufactured.

TABLE 25

IRRADIATED POUCH TEST PACKSeal Strengths - (100°F, 10% R.H. - Ham Pouches)

<u>Structure - Seal Area</u>		<u>Grams/Linear Inch Width (1)</u>			
		<u>Initial (2)</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
<u>50M-50F-.003" Marlex 6050</u>					
Right Seal	Average	5700	3200	4000	5000
	Range	5300-6000+	800-5300	2000-5500	1600-5300
Left Seal	Average	5700	3600	2600	5100
	Range	5300-6000+	800-5300	2500-2700	4500-5500
Bottom Seal	Average	5000	4100	2400	2600
	Range	4800-5400	3500-4500	1500-3100	1800-3500
<u>50M-50F-.002" Nylon 11</u>					
Right Seal	Average	5300	4400	4400	4400
	Range	5000-5600	3200-5000	3500-4800	3800-5100
Left Seal	Average	5300	3800	4400	3800
	Range	5000-5600	3400-4000	3900-4800	3400-4000
Bottom Seal	Average	5100	3800	4000	4300
	Range	4600-5300	3200-4500	3500-4600	4000-4800
<u>50M-50F-.002" Vitel 409</u>					
Right Seal	Average	4400	2300	2800	1400
	Range	3900-4900	700-4500	1100-5100	800-1700
Left Seal	Average	4400	3000	2200	1400
	Range	3900-4900	1900-3500	1100-5100	1100-1650
Bottom Seal	Average	4200	3200	2300	1000
	Range	3100-4900	2900-3800	1400-3500	650-1200

(1) Continental Can Company Test Method STM-13 (Appendix B).

(2) Seals checked on stock at the time pouches were manufactured.

TABLE 26

## IRRADIATED POUCH TEST PACK

Structure-Seal Area	Seal Strengths - (100° F., 90% R.H. - Ham Pouches)					
	Grams/Linear Inch Width (1)					
	Initial*(2) *	1 Week *	3 Months *	6 Months *	12 Months *	
<u>50N-50F-.003"Marlex 6050</u>						
Right Side Seal	Avg. Range	5700 (5300-6000)	4500 (3100-5600)	4300 (1700-5400)	4300 (1900-5700)	5000 (4800-5300)
Left Side Seal	Avg. Range	5700 (5300-6000)	4300 (4200-4600)	4100 (1400-5200)	4500 (3100-5600)	5000 (5000-5000)
Bottom Seal	Avg. Range	5000 (4800-5400)	3900 (3200-4500)	3000 (1500-4100)	2300 (800-4700)	2600 (1400-4300)
<u>50N-50F-.002"Nylon 11</u>						
Right Side Seal	Avg. Range	5300 (5000-5600)	4200 (3600-4900)	4000 (3400-4600)	4100 (3600-4700)	4700 (4200-5000)
Left Side Seal	Avg. Range	5300 (5000-5600)	4600 (4000-5100)	3800 (3500-4300)	4000 (3600-4500)	4300 (4200-5000)
Bottom Seal	Avg. Range	5100 (4600-5300)	4000 (3500-4500)	3700 (2800-4300)	3600 (1800-4600)	4000 (2800-4800)
<u>50M-50F-.002"Vital 409</u>						
Right Side Seal	Avg. Range	4400 (3900-4900)	3400 (2300-4300)	2200 (900-4900)	900 (600-1500)	700 (500-1000)
Left Side Seal	Avg. Range	4400 (3900-4900)	3100 (2300-3900)	2400 (900-5000)	1000 (700-1400)	600 (500-700)
Bottom Seal	Avg. Range	4200 (3100-4900)	4400 (4000-4900)	3000 (2500-3800)	1700 (1400-2300)	600 (500-900)

(1) Continental Can Company Test Method STM-13 (Appendix B).

(2) Seals checked on stock at the time pouches were manufactured.

12.42 Organoleptic Testing (1 week, 3, 6, and 12 months'  
storage) Sensory Difference Tests

The flavor of the ham in the three pouch variables was determined to be "very slightly different" to "slightly different" from that of the irradiated ham in the can control. The range of difference scores for the three package types was 0.22 to 1.80. Average difference scores did not indicate any difference between pouch types in organoleptic performance (See Table 28). Conditions of testing, such as difference in appearance (ham in pouches was more moist than ham in cans) and inherent product variation may have influenced the panelists. A slightly higher percentage of difference judgments was obtained with pouch 50M-50F-.003" Marlex 6050 than was obtained for the other two pouches (See Table 28).

Sensory difference panelists described the ham odor as "smoked," "smoked-burnt," or "burnt" (See Table 29). The ham flavor was generally described in the same terms except for a "fishy" note which was reported as "smoked-fishy" in the control and as "fishy" in the pouch variables (See Table 30). The quality of the irradiated ham from the metal can controls was judged to be "fair" (See Table 27).

"In-Package" Odor Tests

Odor intensity scores recorded at the time of the package opening (prior to microbiological tests) by one person ranged from "0.75" or "trace odor" to "3.75" or "strong odor" for the ham pouch variables. The can control odor intensities ranged from "0" or "no odor"

TABLE 27

IRRADIATED POUCH TEST PACK

ORGANOLEPTIC TESTS

AVERAGE "QUALITY" SCORES (1) FOR HAM IN METAL CANS  
AT VARIOUS TIME PERIODS

SCALE

Excellent = 1

Good = 2

Fair = 3

Poor = 4

<u>Storage Conditions</u>	<u>1 Month</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
75°F - 50% R.H.	3.1	2.7	3.2	2.7
100°F - 10% R.H.	3.2	2.7	2.8	2.7
100°F - 90% R.H.	3.2	2.7	2.9	2.9

- (1) Quality score was the overall rating for odor, taste and texture which was assigned to the control sample by panelists prior to the judging of differences.

TABLE 28

IRRADIATED POUCH TEST PACKS  
ORGANOLEPTIC TEST

Flavor Difference Scores of (1) Ham Pouches vs. metal cans

Pouch Structure	77°F - 50% R.H.			100°F - 10% R.H.			100°F - 90% R.H.					
	1 mo.	3 mo.	6 mo.	12 mo.	1 mo.	3 mo.	6 mo.	12 mo.	1 mo.	3 mo.	6 mo.	12 mo.
50M-50F-.003"Marlex 6050	1.30	0.55	1.22	1.30	1.60	0.89	1.33	1.70	1.40	1.33	1.33	1.10
50M-50F-.002"Nylon 11	1.50	0.22	1.44	0.70	1.10	1.11	1.22	1.80	1.50	1.44	1.67	1.50
50M-50F-.002"Vitel 409	1.20	0.44	1.56	0.90	1.20	0.89	1.56	1.20	0.90	1.11	1.78	1.30

Flavor Difference Scores of (1) Ham Pouches vs. metal cans

Storage Conditions	50M-50F-.003"Marlex 6050	50M-50F-.002"Nylon 11	50M-50F-.002"Vitel 409
77°F - 50% R.H.	1.11	0.97	1.03
100°F - 10% R.H.	1.39	1.32	1.21
100°F - 90% R.H.	1.29	1.53	1.26
	Avg. 1.26	Avg. 1.27	Avg. 1.17

PER CENT OF HAM PANEL FINDING A "FLAVOR DIFFERENCE" (2)

Pouch Structure	50M-50F-.003"Marlex 6050	50M-50F-.002"Nylon 11	50M-50F-.002"Vitel 409
	70.2%	68.4%	66.7%

(1) Difference of pouch sample from metal can control as scored on a 0-5 Scale;  
Average of Scores determined by 10 panelists per evaluation.

(2) Total number of judgments: 342

TABLE 29

IRRADIATED POUCH TEST PACK

ORGANOLEPTIC TEST

SUMMARY OF IRRADIATED HAM "ODOR COMMENTS" (1)

<u>Odor Comments</u>	<u>#3</u>	<u>#4</u>	<u>#8</u>	<u>K</u>
Irradiated Odor:	2	-	-	3
Scorched	1	-	2	4
Smoked-smoky	6	7	7	11
Smoked-burnt	1	4	3	3
Burnt	26	24	23	38
Cooked-smoky	-	1	1	1
V. Slight	1	-	1	4
Slight	8	10	11	15
Moderate	18	12	13	24
Strong	2	3	2	1
Less (than K)	-	3	2	-
More (than K)	7	8	6	-
Pungent (tar-like)	-	-	-	1
Slight	-	-	-	1
More (than K)	1	1	1	-
Appropriate (typical)	-	-	-	19
Non-appropriate (disagreeable)	2	2	2	9
Off (other than irradiated)	2	2	2	-
Sour	2	1	1	-
Medicinal	1	1	1	-
Fishy	-	-	-	1
V. Sl. Fishy	1	-	-	-
Aromatic	-	1	-	-
Spicy	-	-	-	1
Packing house	-	-	1	-
<b>TOTAL NUMBER JUDGMENTS</b>	<b>38</b>	<b>38</b>	<b>38</b>	<b>38</b>

- (1) Ham "odor" comments as made during the sensory difference test.  
 (#3) = 50M-50F-.003" Marlex 6050  
 (#4) = 50M-50F-.002" Nylon 11  
 (#8) = 50M-50F-.002" Vitel 409  
 K = Control Can

TABLE 30  
IRRADIATED POUCH TEST PACK  
ORGANOLEPTIC TESTS

SUMMARY OF IRRADIATED HAM "FLAVOR COMMENTS" (1)

	<u>#3</u>	<u>#4</u>	<u>#8</u>	<u>K</u>
Irradiated Flavor:	2	3	2	1
Scorched	3	1	1	2
Smoked-smoky	3	4	4	9
Smoked-fishy	-	-	-	4
Smoked-burnt	2	1	1	2
Burnt	18	19	15	13
Cooked	21	19	17	25
Cooked-smoky	-	1	-	1
V. Slight	-	2	1	7
Slight	3	1	1	3
Moderate	15	9	11	12
Strong	2	4	2	2
Less (than K)	-	4	1	-
More (than K)	7	4	4	-
Bland-tasteless	-	1	-	9
Appropriate (typical)	3	5	2	16
V. Slight	-	-	-	1
Non-appropriate (disagreeable)	3	2	2	2
Off (other than irradiated)	2	-	-	1
Sour	4	-	1	-
Strong-bitter	-	-	-	1
Medicinal	1	1	1	-
Stale	1	-	-	-
Oxidized	-	-	1	-
Metallic	3	1	1	-
Fishy	2	3	2	-
Fermented	-	-	-	1
Salty	13	14	13	13
Packing house	1	5	2	-
Drier	-	1	1	-
Tough	1	-	-	-
Soft-mushy	1	5	4	-
Greasy	1	1	1	-
TOTAL NUMBER JUDGMENTS	38	38	38	38

(1) Ham "flavor" comments as made during the sensory difference test.  
 (#3) = 50M-50F-.003" Marlex 6050  
 (#4) = 50M-50F-.002" Nylon 11  
 (#8) = 50M-50F-.002" Vitel 409  
 K = Control can

TABLE 31

IRRADIATED POUCH TEST PACKORGANOLEPTIC TESTSSUMMARY "IN-PACKAGE" HAM ODOR INTENSITY SCORES (1)

- 0 = No Off Odor  
 1 = Trace Off Odor  
 2 = Slight Off Odor  
 3 = Moderate Off Odor  
 4 = Strong Off Odor  
 5 = Extremely Strong Off Odor

<u>Storage Conditions</u>	<u>Months</u>	<u>K</u>	<u>#3</u>	<u>#4</u>	<u>#8</u>	<u>Pouch</u>
75°F, 50% R.H.	1	2.00	3.75	3.00	3.75	3.50
	3	1.00	2.25	1.75	2.25	2.08
	6	0	1.75	1.75	2.25	1.92
	12	0	1.25	0.75	1.00	1.00
	Average	0.75	2.25	1.81	2.31	
100°F, 10% R.H.	1	2.00	4.00	3.00	3.75	3.58
	3	1.00	2.00	2.75	3.00	2.58
	6	0	1.00	1.25	0.75	1.00
	12	0	1.25	1.25	1.25	1.25
	Average	0.75	2.06	2.06	2.19	
100°F, 90% R.H.	1	2.00	3.50	3.00	3.25	3.25
	3	1.00	2.50	3.00(2)	2.50(2)	2.67
	6	0	2.75	1.00(2)	1.50(2)	2.08
	12	0	1.00	1.25	1.00	1.08
	Average	0.75	2.44	2.06	2.06	

- (1) Each pouch score represents an average of 4 judgments made by one person (as determined by one person at the time of the initial opening of the pouches three days prior to pouch testing). Each control score represents an average of 2 judgments.
- (2) Musty-moldy odor present.
- (#3) = 50M-50F-.003" Marlex 6050
- (#4) = 50"-50F-.002" Nylon 11
- (#8) = 50M-50F-.002" Vitel 409
- K = Can control

TABLE 32

IRRADIATED POUCH TEST PACK

ORGANOLEPTIC TESTS

SUMMARY "IN-PACKAGE" HAM ODOR COMMENTS (1)

	<u>75°F - 50% R.H.</u>				<u>100°F - 10% R.H.</u>				<u>100°F - 90% R.H.</u>			
Comments	<u>K</u>	<u>3</u>	<u>4</u>	<u>8</u>	<u>K</u>	<u>3</u>	<u>4</u>	<u>8</u>	<u>K</u>	<u>3</u>	<u>4</u>	<u>8</u>
Smoky-woody	-	-	-	-	-	4	1	-	-	-	-	-
Burnt-smoky	1	12	12	12	1	8	12	12	-	-	-	-
Burnt	-	4	4	4	-	4	4	4	-	13	12	12
V. sl. pungent	1	-	-	-	-	-	-	-	-	-	-	-
Tar-like	-	16	16	16	-	12	16	16	-	8	12	12
Sl. tar-like	-	-	-	-	-	-	-	-	-	-	-	-
V. sl. tar-like	2	-	-	-	-	2	-	-	-	-	-	-
More chickeny	-	-	-	-	-	-	-	-	-	-	-	-
Fairly good ham odor	2	-	-	-	2	-	-	-	-	-	-	-
Ham-like	1	-	-	-	-	-	-	1	-	-	-	-
More ham-like	-	-	-	-	-	4	3	-	-	-	2	-
Sl. musty	-	4	4	-	-	-	-	-	-	-	-	-
Strong musty-moldy	-	-	-	-	-	-	-	-	-	5	4	3
Spicy	-	-	-	-	-	-	4	-	-	-	-	-
Sl. putrid	-	-	1	-	-	-	1	-	-	-	1	-
Off-color	-	-	-	-	-	4	4	4	-	4	4	4

Total Number Judgments - 8 for each control (K) and 16 for each pouch variable.

(1) Odor comments as noted by one person at the time of package opening.

(#3) = 50M-50F-.003" Marlex 6050

(#4) = 50M-50F-.002" Nylon 11

(#8) = 50M-50F-.002" Vitel 409

K = Can control

to "2.00" or "slight odor." Small pouch #4 appeared to show lower odor intensities than the other pouches under the 75°F, 50% RH storage conditions, although this trend was not consistent under other storage conditions. Musty-moldy odors were reported present in pouches #4 and #8 under 100°F, 90% RH conditions at 3 and 6 month storage periods. (These odors may have emanated from the outer protective jacket rather than from the inside of the pouch (See Table 31).) Comments made during the "In-Package" sniff tests indicated that the character of the ham odor was more "burnt" under the 100°F, 90% RH conditions than it was under the other storage conditions. The ham also showed fewer "tar-like" comments at the 100°F, 90% RH conditions than it did under the other storage conditions (See Table 32).

#### 12.5 Chicken Pouch Test Pack

##### Chicken Specification

A boneless chicken roll was prepared as described in Appendix G. This roll was cut into slices 1/2" and trimmed on two sides to make a 3" x 4-1/4" chicken slice.

#### 12.51 Physical Packaging Tests (1 week, 3, 6, and 12 months' storage)

##### Weight Check

Weight checks were made at the 3, 6, and 12 months' periods as described in Section 12.3, Package Evaluation Procedures. All weight checks on all packages were less than 0.1% product weight change which was considered to be an insignificant weight loss. Results of this evaluation are reported in Table 33.

#### Head Space Gas

Since there was not sufficient head space for regular analysis, the amount of retained vacuum in the packages was tested by Continental Can Test Method STM-181 (See Appendix L). The results of this analysis are reported in Table 35. These results show a consistent retained vacuum after the 12 months' storage period. This effect appears to be similar for all products under all storage conditions, thus indicating no preference in regards to the package type on vacuum retention with this product.

Due to a mix-up in the initial inspection and lack of sufficient additional can controls, the 1 week head space analysis on the chicken can controls was not conducted. However, at the 3 months inspection period, the vacuum in the cans was checked and found to vary from 0 to 10" of vacuum in the cans indicating a variation in the samples as prepared. At the 6 and 12 months' period, a total head space gas analysis was run on the can controls by chromatography analysis and the vacuum checked by the double expansion technique and found to be variable. Since the head space gas analysis appeared to be similar on the samples with the high and low vacuum, it was concluded that this variation in vacuum in the cans was due to the initial closing techniques. The oxygen in the head space at the 12 months' period is essentially 0% with the reported oxygen and argon equal to 0.6% to 0.7% being primarily argon. The results of the can control head space analysis are reported in Table 34.

TABLE 33

IRRADIATED POUCH TEST PACK

Product Weight Loss - (Chicken Pouches)

	Percent Product Weight Loss				
<u>Structure - Storage</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>		
			<u>Average</u>	<u>Range</u>	
<u>77°F, 50% R.H.</u>					
50M-50F-.003" Marlex 6050	<0.1	<0.1	0.04	(0.04-0.05)	
50M-50F-.002" Nylon 11	<0.1	<0.1	0.07	(0.07-0.08)	
50M-50F-.002" Vitel 409	<0.1	<0.1	0.04	(0.03-0.04)	
<u>100°F, 10% R.H.</u>					
50M-50F-.003" Marlex 6050	<0.1	<0.1	0.02	(0 -0.03)	
50M-50F-.002" Nylon 11	<0.1	<0.1	0.04	(0 -0.09)	
50M-50F-.002" Vitel 409	<0.1	<0.1	0.01	(0 -0.02)	
<u>100°F, 90% R.H.</u>					
50M-50F-.003" Marlex 6050	<0.1	<0.1	0.08	(0.06-0.09)	
50M-50F-.002" Nylon 11	<0.1	<0.1	0.1	(0.06-0.1 )	
50M-50F-.002" Vitel 409	<0.1	<0.1	0.05	(0.03-0.06)	

TABLE 34

IRRADIATED POUCH TEST PACKHead Space Gas Analysis - (Canned Chicken Controls)

<u>GAS</u> Sample No.	<u>3 Months</u>		<u>6 Months</u>		<u>12 Months</u>	
	1	2	1	2	1	2
<u>77°F, 50% R.H.</u>						
CO <sub>2</sub>	-	-	-	8.1	6.3	7.9
H <sub>2</sub>	-	-	-	32.5	25.0	45.4
O <sub>2</sub> + A	-	-	-	0.6	0.9	0.6
N <sub>2</sub>	-	-	-	51.0	65.5	43.0
CO	-	-	-	6.7	1.2	1.3
CH <sub>4</sub>	-	-	-	1.1	1.1	1.8
Total Per Cent	-	-	-	100.0	100.0	100.0
Total Head Space Volume (ML)	-	-	-	125.8	128.5	75.8
Vacuum in Can on Opening (inches)	-	-	-	10		
<u>100°F, 10% R.H.</u>						
CO <sub>2</sub>	-	-	6.7	8.2	(1)	
H <sub>2</sub>	-	-	36.0	35.2	(1)	
O <sub>2</sub> + A	-	-	0.7	0.6		
N <sub>2</sub>	-	-	49.0	48.0		
CO	-	-	6.3	6.7		
CH <sub>4</sub>	-	-	1.3	1.3		
Total Per Cent	-	-	100.0	100.0		
Total Head Space Volume (ML)	-	-	57.0	102.0		
Vacuum in Can on Opening (inches)	-	9.5	0	8		

(1) No Samples Available

### Bond Strengths

The bond strengths were checked in the seal areas and body areas between the foil and food contacting films by using Continental Can Test Method STM-8 (Appendix A). The results of the bond determinations are reported in Tables 36 through 41. The bond strengths showed a decrease after 1 week storage from the initial values, both in the body and seal areas. The bond strength continued to decrease after the 1 week through to the 3 months' period and then remained essentially the same from the 3 months' storage through the 12 months' storage under most conditions. The 50M-50F-.003" Marlex 6050 showed lowering of bond at the 12 months' period under the 100°F, 10% RH, and 100°F, 90% RH storage conditions, probably due to the fat from the food product permeating the film at this elevated temperature at the 12 months' period. However, bond was still maintained and no delamination was encountered. The 50M-50F-.002" Vitel 409 developed film embrittlement after the 12 months' storage, which brittleness was not evident at the 6 months' storage period. This embrittlement did not permit bond determinations after storage at 100°F, 10% RH. Storage at the 100°F, 90% RH caused bond lowering, which again, is apparently due to the brittleness of the film. Only the bonds on the 50M-50F-.003" Marlex 6050 appear to be critical at the 100°F, 10% RH and 100°F, 90% RH at the 12 months' storage period in the body area of the pouches. However, since bonds were still maintained in the seal areas, it was concluded that this

TABLE 35

Irradiated Pouch Test PackInches of Vacuum in Chicken Pouches

<u>Storage and Structure</u>	<u>1 Week</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
<u>77°F., 50% R.H.</u>				
50M-50F-.003"Marlex 6050	Avg. Range 6.9 (6.3-7.2)	5.3 (3.5-6.7)	5.2 (2.9-6.3)	5.7 (5.1-5.9)
50M-50F-.002"Nylon 11	Avg. Range 6.0 (4.8-7.2)	5.5 (5.1-5.9)	4.7 (3.9-5.5)	4.9 (4.7-5.1)
50M-50F-.002"Vitel 409	Avg. Range 5.2 (4.2-6.7)	4.1 (4.0-4.4)	4.2 (3.2-5.9)	4.5 (3.5-5.1)
<u>100°F., 10% R.H.</u>				
50M-50F-.003"Marlex 6050	Avg. Range 8.2 (5.5-9.9)	7.0 (5.5-8.3)	5.6 (4.7-6.3)	7.4 (4.3-10.2)
50M-50F-.002"Nylon 11	Avg. Range 5.8 (4.2-7.2)	4.9 (3.2-7.1)	4.2 (3.5-4.7)	5.8 (4.3-7.1)
50M-50F-.002"Vitel 409	Avg. Range 5.6 (4.2-6.7)	5.3 (4.0-7.5)	4.6 (3.2-5.9)	5.9 (4.3-7.1)
<u>100°F., 90% R.H.</u>				
50M-50F-.003"Marlex 6050	Avg. Range 7.9 (7.2-9.1)	10.1 (9.5-13.0)	6.3 (4.7-7.1)	11.0 (8.2-12.6)
50M-50F-.002"Nylon 11	Avg. Range 6.8 (4.2-9.1)	6.7 (4.2-7.9)	4.1 (2.8-5.5)	6.2 (5.5-7.8)
50M-50F-.002"Vitel 409	Avg. Range 6.3 (4.8-7.5)	7.4 (5.1-10.2)	4.6 (3.9-5.5)	10.7 (8.6-11.7)

TABLE 36

## IRRADIATED POUCH TEST PACK

Body Area Bond Strengths - (77° F., 50% R.H. - Chicken Pouches)

Structure - Body Area (1)	Initial *	(2) 1 Week *	Grams/Linear Inch Width (3)		
			3 Months *	6 Months *	12 Months *
50M-50F-.003"Marlex 6050	Avg. Range	910 (850-950)			
Right Side Area	Avg. Range	630 (540-800)	400 (370-430)	480 (450-550)	390 (260-400)
Left Side Area	Avg. Range	530 (390-780)	390 (350-470)	440 (350-550)	380 (330-450)
Bottom Area	Avg. Range	650 (400-920)	370 (350-400)	440 (400-450)	410 (400-450)
50M-50F-.002"Nylon 11	Avg. Range	560 (560-560)			
Right Side Area	Avg. Range	460 (310-670)	340 (280-400)	390 (350-470)	340 (320-350)
Left Side Area	Avg. Range	430 (290-650)	310 (280-350)	370 (320-450)	350 (280-470)
Bottom Area	Avg. Range	380 (280-550)	310 (300-320)	360 (300-400)	300 (250-350)
50M-50F-.002"Vitel 409	Avg. Range	593 (510-650)			
Right Side Area	Avg. Range	650 (500-790)	460 (430-500)	580 (550-620)	640 (450-700)
Left Side Area	Avg. Range	580 (480-790)	460 (400-500)	650 (450-850)	610 (400-850)
Bottom Area	Avg. Range	560 (500-610)	410 (400-450)	640 (540-800)	650 (600-700)

(1) Body areas between the foil and food contacting film.

(2) Bonds checked on stock at the time pouches were manufactured.

(3) Continental Can Company Test Method STM-8 (Appendix A).

TABLE 37

IRRADIATED POUCH TEST PACK

body Area Bond Strengths - (100° F., 10% R.H. - Chicken Pouches)

Structure - Body Area (1)	Initial *	(2) *	Grams/Linear Inch Width (3)		
			1 Week *	3 Months *	6 Months *
<u>50M-50F-.003"Marlex 6050</u>	Avg. Range	910 (850-950)			
Right Side Area	Avg. Range		430 (350-520)	420 (370-480)	520 (400-600)
Left Side Area	Avg. Range		500 (400-600)	390 (340-470)	520 (450-650)
Bottom Area	Avg. Range		470 (420-550)	350 (300-400)	500 (450-550)
<u>50M-50F-.002"Nylon 11</u>	Avg. Range	560 (560-560)			
Right Side Area	Avg. Range		490 (380-580)	290 (280-320)	380 (320-450)
Left Side Area	Avg. Range		380 (270-580)	280 (250-300)	380 (320-450)
Bottom Area	Avg. Range		340 (250-500)	270 (200-280)	480 (320-750)
<u>50M-50F-.002"Vitel 409</u>	Avg. Range	593 (510-650)			
Right Side Area	Avg. Range		540 (420-720)	470 (460-480)	450 (400-530)
Left Side Area	Avg. Range		640 (500-800)	470 (440-550)	450 (400-550)
Bottom Area	Avg. Range		550 (400-700)	450 (430-470)	490 (400-550)

(1) Body areas between the foil and food contacting film.

(2) Bonds checked on stock at the time pouches were manufactured.

(3) Continental Can Company Test Method STM-8 (Appendix A).

TABLE 38

IRRADIATED POUCH TEST PACK

Body Area Bond Strengths - (100° F., 90% R.H. - Chicken Pouches)

Structure - Body Area (1)	Initial * (2)	Grams/Linear Inch Width (3)			
		1 Week *	3 Months *	6 Months *	12 Months *
<u>50N-50F-.003"Marlex 6050</u>	Avg. Range	910 (850-950)			
Right Side Area	Avg. Range	470 (400-540)	480 (380-670)	510 (450-600)	170 ( 80-250)
Left Side Area	Avg. Range	530 (400-750)	400 (350-450)	530 (450-650)	160 (150-200)
Bottom Area	Avg. Range	420 (340-500)	390 (350-450)	490 (450-500)	150 ( 0-200)
<u>50N-50F-.002"Nylon 11</u>	Avg. Range	560 (560-560)			
Right Side Area	Avg. Range	470 (320-730)	380 (350-400)	400 (300-500)	230 (150-300)
Left Side Area	Avg. Range	510 (350-850)	390 (280-470)	390 (300-480)	230 (150-350)
Bottom Area	Avg. Range	400 (350-450)	280 (250-300)	350 (300-400)	250 (200-350)
<u>50N-50F-.002"Vitel 409</u>	Avg. Range	593 (510-650)			
Right Side Area	Avg. Range	430 (320-500)	520 (430-750)	770 (650-900)	430 (350-500)
Left Side Area	Avg. Range	500 (370-700)	520 (420-700)	690 (500-850)	360 (300-450)
Bottom Area	Avg. Range	380 (300-450)	520 (400-590)	580 (520-700)	370 (350-400)

(1) Body areas between the foil and food contacting film.

(2) Bonds checked on stock at the time pouches were manufactured.

(3) Continental Can Company Test Method STM-8 (Appendix A).

TABLE 39

IRRADIATED POUCH TEST PACKSeal Area Bond Strength - (77°F, 50% R.H. - Chicken Pouches)

<u>Structure - Seal Areas (2)</u>		<u>Grams/Linear Inch Width (1)</u>			
		<u>1 Week</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
		*	*	*	*
<u>50M-50F-.003" Marlex 6050</u>					
Right Seal Area	Average	1000	470	440	425
	Range	(980-1000+)	(450-480)	(400-500)	(400-500)
Left Seal Area	Average	960	460	380	430
	Range	(700-1000+)	(380-520)	(300-500)	(350-500)
Bottom Seal Area	Average	940	450	390	450
	Range	(800-1000 )	(400-500)	(350-400)	(400-500)
<u>50M-50F-.002" Nylon 11</u>					
Right Seal Area	Average	910	460	340	410
	Range	(770-1000+)	(400-550)	(300-450)	(350-450)
Left Seal Area	Average	950	390	320	390
	Range	(800-1000+)	(350-450)	(300-400)	(300-550)
Bottom Seal Area	Average	970	380	300	340
	Range	(880-1000+)	(350-400)	(300-320)	(300-350)
<u>50M-50F-.002" Vitel 409</u>					
Right Seal Area	Average	850	540	540	650
	Range	(800-940 )	(500-550)	(500-620)	(450-750)
Left Seal Area	Average	860	520	630	650
	Range	(670-970 )	(450-600)	(450-830)	(450-850)
Bottom Seal Area	Average	760	500	610	690
	Range	(630-850 )	(430-550)	(500-780)	(650-750)

- (1) Continental Can Company Test Method STM-8 (Appendix A).  
 (2) Seal areas between the foil and food contacting film.

TABLE 40

IRRADIATED POUCH TEST PACKSeal Area Bond Strength - (100°F, 10% R.H. - Chicken Pouches)

		<u>Grams/Linear Inch Width (1)</u>			
<u>Structure - Seal Area (3)</u>		<u>1 Week</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
		*	*	*	*
<u>50M-50F-.003" Marlex 6050</u>					
Right Seal Area	Average	760	490	540	375
	Range	(570-1000 )	(450-550)	(410-750)	(350-400)
Left Seal Area	Average	860	490	480	360
	Range	(680-1100 )	(420-550)	(350-780)	(300-500)
Bottom Seal Area	Average	830	450	440	340
	Range	(750-950 )	(420-550)	(370-500)	(250-400)
<u>50M-50F-.002" Nylon 11</u>					
Right Seal Area	Average	980	410	340	350
	Range	(920-1000+)	(340-440)	(280-400)	(250-450)
Left Seal Area	Average	880	380	340	370
	Range	(800-1000+)	(320-420)	(300-400)	(350-400)
Bottom Seal Area	Average	950	430	430	350
	Range	(820-1000+)	(400-480)	(280-700)	(300-400)
<u>50M-50F-.002" Vitel 409</u>					
Right Seal Area	Average	810	540	410	(2)
	Range	(730-880 )	(520-550)	(400-450)	
Left Seal Area	Average	830	570	410	(2)
	Range	(700-970 )	(500-700)	(350-500)	
Bottom Seal Area	Average	740	520	450	(2)
	Range	(700-800 )	(500-550)	(400-500)	

- (1) Continental Can Company Test Method STM-8 (Appendix A).  
 (2) Vitel 409 film too brittle to permit bond determination.  
 (3) Seal areas between the foil and food contacting film.

TABLE 41

IRRADIATED POUCH TEST PACKSeal Area Bond Strength - (100°F, 90% R.H. - Chicken Pouches)

<u>Structure - Seal Area (2)</u>		<u>Gr    Linear Inch Width (1)</u>			
		<u>1 Week</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
		*	*	*	*
<u>50M-50F-.003" Marlex 6050</u>					
Right Seal Area	Average	750	440	450	200
	Range	(740-800)	(350-520)	(390-500)	(150-250)
Left Seal Area	Average	830	470	480	200
	Range	(700-950)	(410-550)	(400-600)	(150-300)
Bottom Seal Area	Average	710	440	420	210
	Range	(540-800)	(420-450)	(400-450)	(100-250)
<u>50M-50F-.002" Nylon 11</u>					
Right Seal Area	Average	830	410	330	270
	Range	(750-920)	(300-450)	(280-450)	(200-380)
Left Seal Area	Average	810	400	350	260
	Range	(670-980)	(320-480)	(260-460)	(200-350)
Bottom Seal Area	Average	760	310	290	290
	Range	(650-820)	(280-450)	(250-350)	(200-350)
<u>50M-50F-.002" Vitel 409</u>					
Right Seal Area	Average	750	520	720	440
	Range	(640-850)	(450-650)	(650-800)	(350-500)
Left Seal Area	Average	760	550	610	360
	Range	(600-890)	(450-630)	(480-800)	(300-450)
Bottom Seal Area	Average	740	530	520	380
	Range	(650-830)	(450-620)	(480-640)	(350-400)

(1) Continental Can Company Test Method STM-8 (Appendix A).

(2) Seal areas between the foil and food contacting film.

pouch was still functional after this 12 months' storage period. It is felt that all of the bond strengths are sufficiently high for a functional package.

#### Seal Strengths

The seal strengths were checked on the side and bottom seals (both film directions) by using Continental Can Company's Test Method STM-13 (Appendix R). The results of the seal strength tests are reported in Tables 42 through 44. The seal strength values lowered on storage. However, they appeared to be stable for the 50M-50F-.002" Nylon 11 at the 3, 6, and 12 months' storage period, showing good functional level through this 12 month storage period. The 50M-50F-.003" Marlex 6050 showed a lowering of initial seal strengths in the bottom seal area at the 1 week and gradually decreasing through the 12 month storage period. This lowering of the bottom seal apparently was due to the linear nature of the polymeric film. However, it was still maintained sufficiently high to be functional after the 12 months' storage period. The seal strengths in the side seals appear to be fairly constant throughout the test exposure on this 50M-50F-.003" Marlex 6050. The 50M-50F-.002" Vitel 409 showed satisfactory seal maintenance through 6 months at 77°F, 50% RH. However, at the end of the 12 months' period, it showed some lowering of the seals at this storage condition and at 100°F, 10% RH and 100°F, 90% RH showed considerable degradation, starting at the 3 months' period and lowering to unsatisfactory levels. This was apparently due to embrittlement of the Vitel 409 polymer on storage as

TABLE 42

IRRADIATED POUCH TEST PACK

Seal Strengths -(77° F., 50% R.H. - Chicken Pouches)

Structure - Seal Area	Initial (2) *	Grams/Linear Inch Width (1)			
		1 Week *	3 Months *	6 Months *	12 Months *
<u>504-50F-.003"Marlex 6050</u>					
Right Side Seal	Avg. Range 5700 (5300-6000f)	5400 (4000-6000f)	5300 (3900-6000f)	5400 (4300-6000f)	5500 (4500-6000f)
Left Side Seal	Avg. Range 5700 (5300-6000f)	6000 (6000f-6000f)	5300 (4500-6000f)	5500 (4900-6000f)	5800 (5000-6000f)
Bottom Seal	Avg. Range 5000 (4800-5400)	4200 (2200-5900)	3800 (2800-5200)	4200 (3000-5000)	3400 (1950-4400)
<u>50M-50F-.002"Nylon 11</u>					
Right Side Seal	Avg. Range 5300 (5000-5600)	4800 (3900-5300)	4600 (4200-4900)	4500 (4100-5100)	4600 (4000-5200)
Left Side Seal	Avg. Range 5300 (5000-5600)	4900 (4200-5300)	4600 (4000-5200)	4100 (3500-5100)	4500 (3800-5000)
Bottom Seal	Avg. Range 5100 (4600-5300)	4300 (3200-5400)	4000 (3000-4900)	4000 (3100-4800)	3600 (2500-4400)
<u>50M-50F-.002"Vitel 409</u>					
Right Side Seal	Avg. Range 4400 (3900-4900)	4500 (3800-4800)	4600 (4200-5000)	4100 (2400-5100)	4200 (1800-4900)
Left Side Seal	Avg. Range 4400 (3900-4900)	4400 (3400-5200)	4500 (4000-4800)	4100 (3800-4400)	3800 (1300-5400)
Bottom Seal	Avg. Range 4200 (3100-4900)	4300 (3600-4500)	4000 (3600-4500)	4200 (3400-4800)	4500 (3600-5100)

(1) Continental Can Company Test Method STM-13 (Appendix B).

(2) Seals checked on stock at the time pouches were manufactured.

TABLE 43

IRRADIATED POUCH TEST PACK

Seal Strengths - (100° F., 10% R.H. - Chicken Pouches)

Structure - Seal Area	Initial (2) *	Grams/Linear Inch Width (1)				
		1 Week *	3 Months *	6 Months *	12 Months *	
<u>50M-50F-.003"Marlex 6050</u>						
Right Side Seal	Avg. Range	5700 (5300-6000f)	5900 (5500-6000f)	5300 (4200-6000f)	5600 (3100-6000f)	5800 (5300-6000f)
Left Side Seal	Avg. Range	5700 (5300-6000f)	5100 (4100-6000f)	5500 (5300-6000f)	5900 (5800-6000f)	6000f (6000f-6000f)
Bottom Seal	Avg. Range	5000 (4800-5400)	4300 (2300-5500)	4400 (2900-5400)	3900 (2500-5000)	3600 (2600-4700)
<u>50M-50F-.002"Nylon 11</u>						
Right Side Seal	Avg. Range	5300 (5000-5600)	4700 (4200-5400)	4400 (4100-4800)	4300 (2800-4500)	4600 (4300-4800)
Left Side Seal	Avg. Range	5300 (5000-5600)	4500 (4100-4800)	4400 (4100-4800)	4200 (3300-4600)	4500 (3800-5100)
Bottom Seal	Avg. Range	5100 (4600-5300)	4400 (3600-5500)	3700 (2500-4700)	3200 (2600-3600)	4500 (3600-4800)
<u>50M-50F-.002"Vitel 409</u>						
Right Side Seal	Avg. Range	4400 (3900-4900)	4500 (3700-5100)	3000 (1400-4600)	1800 (500-3400)	1400 (700-2000)
Left Side Seal	Avg. Range	4400 (3900-4900)	4400 (4000-5500)	2600 (700-3800)	2900 (700-4500)	2400 (1200-1800)
Bottom Seal	Avg. Range	4200 (3100-4900)	4600 (4100-5200)	3500 (2600-3800)	2600 (1800-3600)	4500 (3600-5100)

(1) Continental Can Company Test Method STM-13 (Appendix B).

(2) Seals checked on stock at the time pouches were manufactured.

TABLE 44

IRRADIATED POUCH TEST PACK

Seal Strengths - (100° F., 90% R.H. - Chicken Pouches)

Structure - Seal Area		Grams/Linear Inch Width (1)				
		Initial *	1 Week *	3 Months *	6 Months *	12 Months *
50H-50F-.003"Marlex 6050						
Right Side Seal	Avg. Range	5700 (5300-6000f)	5000 (3400-5900)	4400 (1400-6000f)	5600 (4800-6000f)	5800 (5100-6000f)
Left Side Seal	Avg. Range	5700 (5300-6000f)	5100 (4700-5000f)	4500 (1800-5600)	5900 (4800-6000f)	5700 (5100-6000f)
Bottom Seal	Avg. Range	5000 (4800-5400)	3900 (2500-5200)	3800 (2100-5400)	3700 (1800-5300)	3200 (2300-4400)
50H-50F-.002"Nylon 11						
Right Side Seal	Avg. Range	5300 (5000-5600)	4700 (4100-5400)	3900 (2700-5000)	4000 (3300-4500)	4500 (3900-5200)
Left Side Seal	Avg. Range	5300 (5000-5600)	4400 (4100-4600)	4500 (3900-5000)	4000 (3300-4600)	4400 (4100-5100)
Bottom Seal	Avg. Range	5100 (4600-5300)	4000 (2700-5100)	3900 (2800-4500)	3200 (2300-4400)	3500 (2700-4700)
50H-50F-.002"Vitel 409						
Right Side Seal	Avg. Range	4400 (3900-4900)	4700 (3700-5500)	1300 ( 700-1700)	1300 ( 900-1700)	1400 ( 500-3800)
Left Side Seal	Avg. Range	4400 (3900-4900)	4200 (3900-4400)	1300 ( 800-1800)	1300 (1000-1500)	1800 ( 600-4700)
Bottom Seal	Avg. Range	4200 (3100-4900)	4600 (4100-5400)	3300 (2700-4500)	2000 (1100-2900)	910 ( 600-2800)

(1) Continental Can Company Test Method STM-13 (Appendix B).

(2) Seals checked on stock at the time pouches were manufactured.

was evident by the embrittlement in the checking of the samples for seal and bond at these conditions. Thus, only the 50M-50F-.002" Vitel 409 would be considered unsatisfactory from a seal strength standpoint after the 12 months' storage period.

#### Observations

There was no noted delamination or other package changes observed in the chicken pouches other than the reported decreased variations in bond and seal strength and film embrittlement noted on the 50M-50F-.002" Vitel 409 laminate.

#### 12.52 Organoleptic Testing (1 week, 3, 6, and 12 months storage)

##### Sensory Difference Tests

The flavor of the irradiated chicken in the three pouch variables was determined to be "very slightly different" from that of the irradiated chicken in the can control. The range of difference scores for the three package types was 0.30 to 1.44 (See Table 46). Average difference scores did not indicate any difference in organoleptic performance between the three pouch types.

A higher percentage of difference judgments was obtained with pouch 50M-50F-.003" Marlex 6050 than was obtained for the other two pouches (See Table 46). This difference was consistent with a trend indicated in the irradiated ham study. Sensory difference panelists described the chicken odor as "scorched," "smoked" or "burnt" (See Table 47). The chicken flavor was generally described as "burnt" or "cooked"

(See Table 48). The overall quality of the irradiated chicken from the metal can controls was judged to be "fair," on a scale of "excellent," "good," "fair," and "poor" quality (See Table 45).

#### "In-Package" Odor Tests

Odor intensity scores recorded at the time of package opening by one person ranged from "1.25" or "slight" to "4.00" or "strong" for the chicken pouch variables. The can control intensity scores ranged from "1.00" or "trace" to "2.50" or "slight" to "moderate." Chicken intensity odor scores tended to be lower in the pouch samples stored at 100°F, 90% RH than in the pouch samples stored at 100°F, 10% RH or 75°F, 50% RH.

Musty-moldy odors were reported present in pouches 3 and 4 under 100°F, 90% RH conditions at 3 and 6 months' storage periods. (As in the irradiated ham, these odors may have emanated from the outer protective jacket rather than from the inside of the pouch (See Table 49).)

Comments made during the "in-package" sniff tests indicated that the character of the chicken odor was usually described as "burnt-smoky" or "burnt," with fewer "tar-like" comments occurring at the lower storage temperatures (See Table 50).

#### 12.6 Bacon Test Pack

##### Bacon Specification

The bacon used was of a commercial, Grade A, uncooked product,

TABLE 45

IRRADIATED POUCH TEST PACK

ORGANOLEPTIC TESTS

AVERAGE "QUALITY" SCORES FOR CHICKEN IN METAL CANS  
AT VARIOUS TIME PERIODS (1)

SCALE

Excellent = 1

Good = 2

Fair = 3

Poor = 4

<u>Storage Conditions</u>	<u>1 Month</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
75°F - 50% R.H.	3.0	3.2	3.3	3.1
100°F - 10% R.H.	3.3	2.8	3.3	3.1
100°F - 90% R.H.	3.3	3.2	3.3	3.1

(1) Evaluated by panelists prior to judging "differences."

TABLE 46

IRRADIATED POUCH TEST PACKS  
ORGANOLEPTIC TESTS

Flavor difference scores of (1) Chicken Pouches vs. metal cans

Fouch Types	77°F. - 50% R.H.				100°F. - 10% R.H.				100° F. - 90% R.H.			
	1 mo.	3 mo.	6 mo.	12 mo.	1 mo.	3 mo.	6 mo.	12 mo.	1 mo.	3 mo.	6 mo.	12 mo.
50M-50F-.003"Marlex 6050	0.44	0.73	0.78	0.50	0.33	0.44	1.11	0.60	0.44	0.67	0.67	0.80
50M-50F-.002"Nylon 11	0.33	1.44	0.44	0.70	0.44	0.44	0.56	0.50	0.44	1.00	0.56	0.50
50M-50F-.002"Vital 409	0.33	0.56	0.67	0.50	0.41	0.56	0.67	0.30	0.44	0.22	0.67	1.10

Flavor Difference Scores of (1) Chicken Pouches vs. metal cans

Storage Conditions	50M-50F-.003"Marlex 6050	50M-50F-.002"Nylon 11	50M-50F-.002"Vital 409
77° F. - 50% R.H.	0.62	0.73	0.51
100° F. - 10% R.H.	0.62	0.51	0.46
100° F. - 90% R.H.	0.65	0.62	0.62
	Avg. 0.63	Avg. 0.62	Avg. 0.53

PER CENT OF CHICKEN PANEL FINDING A "FLAVOR DIFFERENCE" (2)

Pouch Structure	50M-50F-.003"Marlex 6050	50M-50F-.002"Nylon 11	50M-50F-.002"Vital 409
	45.0%	41.4%	32.4%

(1) Difference of pouch sample from metal can control as scored on a 0-5 Scale.

(2) Total number of judgements: 333

TABLE 47  
IRPADIATED POUCH TEST PACK  
ORGANOLEPTIC TESTS

SUMMARY OF IRRADIATED CHICKEN "ODOR" COMMENTS (1)

<u>Odor Comments</u>	<u>#3</u>	<u>#4</u>	<u>#8</u>	<u>K</u>
Irradiated odor:	-	-	-	9
Scorched	1	2	2	9
Smoked-smoky	2	1	3	6
Burnt	17	13	14	39
Cooked	-	-	-	2
Cooked-smoky				
None	2	1	1	1
V. Slight	-	-	-	6
Slight	4	-	2	9
Moderate	6	6	4	26
Strong	1	1	2	9
Less (than K)	3	5	-	-
More (than K)	5	1	5	-
Pungent (tar-like)				
Slight	-	-	-	1
Less (than K)	-	1	-	-
Milder (than K)	-	1	1	-
Bland-odorless	-	-	1	5
Appropriate (typical)	1	1	1	9
None	-	1	-	-
Non-appropriate (disagreeable)	-	-	-	4
Slight	1	-	-	-
Off (other than irradiated)	1	-	1	-
Sour	-	2	2	-
Musty	-	-	-	1
Stale	1	-	-	-
Skunky	2	-	1	-
Fishy	-	-	-	1
Sl. putrid	-	1	1	1
Packing mouse	1	2	-	1
TOTAL NUMBER JUDGMENTS	37	37	37	37

(1) Chicken "odor" comments as made during the sensory difference test.

(#3) = 50M-50F-.003" Marlex 6050

(#4) = 50M-50F-.002" Nylon 11

(#8) = 50M-50F-.002" Vitel 409

K = Can control

TABLE 48  
IRRADIATED POUCH TEST PACK  
ORGANOLEPTIC TESTS

SUMMARY OF IRRADIATED CHICKEN "FLAVOR" COMMENTS (1)

<u>Odor Comments</u>	<u>#3</u>	<u>#4</u>	<u>#8</u>	<u>K</u>
Irradiated flavor:	1	2	1	1
Scorched	1	-	-	-
Smoked-smoky	-	-	-	2
Smoked-fishy	-	-	-	-
Smoked-burnt	-	-	-	1
Burnt	12	8	8	19
Cooked	10	5	6	16
Cooked-smoky	-	-	-	-
None	-	-	-	5
V. Slight	-	-	-	1
Slight	1	-	-	5
Moderate	4	1	3	16
Strong	2	2	1	-
Less (than K)	2	2	-	-
More (than K)	7	4	5	-
Weaker than odor	-	-	-	1
Bland-tasteless	1	-	-	14
Appropriate (typical)	-	2	-	2
V. Slight	-	-	-	1
Non-appropriate (disagreeable)	1	-	1	3
Slight	1	2	1	-
Off (other than irradiated)	1	-	-	-
Sour	-	-	1	-
Bitter	-	1	-	-
Medicinal	1	-	-	-
Stale	-	-	-	1
Metallic	2	1	-	-
Salty	-	-	-	2
Packing house	1	1	1	5
Dry	1	1	1	2
TOTAL NUMBER JUDGMENTS	37	37	37	37

(1) Chicken "flavor" comments as made during the sensory difference test.

(#3) = 50M-50F-.003" Marlex 6050

(#4) = 50M-50F-.002" Nylon 11

(#8) = 50M-50F-.002" Vitel 40<sup>n</sup>

K = Can control

TABLE 49

IRRADIATED POUCH TEST PACKORGANOLEPTIC TESTSSUMMARY OF ODOR INTENSITY SCORES (1)CHICKEN "IN-PACKAGE" ODOR TESTS

0 = No Off Odor

1 = Trace Off Odor

2 = Slight Off Odor

3 = Moderate Off Odor

4 = Strong Off Odor

5 = Extremely Strong Off Odor

Storage Conditions

<u>Product</u>	<u>°F/% Humidity</u>	<u>Months</u>	<u>Can Control</u>	(a) <u>Pouch 3</u>	(b) <u>Pouch 4</u>	(c) <u>Pouch 8</u>	<u>Average (Pouches Only)</u>
Chicken	75/50	1	1.00	3.25	3.00	2.50	2.92
		3	1.00	3.00	3.00	2.50	2.92
		6	1.50	3.50	3.00	3.75	3.42
		12	1.00	3.75	2.75	2.50	3.00
		Average	1.13	3.38	2.94	2.81	
	100/10	1	2.00	4.00	2.75	2.50	3.08
		3	1.00	2.50	2.50	2.75	2.58
		6	1.50	2.75	3.75	2.50	3.00
		12	2.50	4.00	2.50(3)	2.50	3.00
		Average	1.75	3.31	2.88	2.56	
	100/90	1	2.00	2.25	3.00	1.25	2.17
		3	1.00	2.00(2)	1.75(2)	2.75	2.17
		6	1.50	2.75(2)	3.25(1)	2.75	2.25
		12	2.50	2.25	3.00	2.25	2.83
		Average	1.75	2.31	2.75	2.25	

- (1) Each pouch score represents an average of 4 judgments made by one person; each control score represents an average of 2 judgments.  
 (2) Musty-moldy odor present.  
 (3) Inside of one pouch (out of four) was mottled; product showed off-color and off-odor.  
 (a) 50M-50F-.003" Marlex 6050  
 (b) 50M-50F-.002" Nylon 11  
 (c) 50M-50F-.002" Vitel 409

TABLE 50

IRRADIATED POUCH TEST PACKORGANOLEPTIC TESTSSUMMARY OF ODOR COMMENTS (1)CHICKEN "IN-PACKAGE" ODOR TESTS

	<u>75°F-50% R.H.</u>				<u>100°F-10% R.H.</u>				<u>100°F-90% R.H.</u>			
<u>Comments:</u>	<u>K</u>	<u>3</u>	<u>4</u>	<u>8</u>	<u>K</u>	<u>3</u>	<u>4</u>	<u>8</u>	<u>K</u>	<u>3</u>	<u>4</u>	<u>8</u>
Smoky												
Sl. smoky	-	-	-	-	-	-	-	-	-	4	-	-
V. sl. smoky	3	-	-	-	3	-	-	-	-	-	-	4
Burnt-smoky	5	8	12	12	5	8	12	12	-	8	8	8
Burnt	2	4	4	4	2	4	4	4	-	4	4	4
Pungent	-	4	-	-	-	4	-	-	-	-	4	-
Sl. pungent	-	-	4	4	-	-	4	4	-	-	-	-
Tar-like	8	12	4	4	8	16	12	12	-	12	16	12
More chickeny	-	-	-	-	-	-	-	-	-	4	-	4
Off	-	-	-	-	-	-	1(2)	-	-	-	-	-
Musty-moldy	-	-	-	-	-	-	-	-	-	-	1	-
Sl. musty-moldy	-	-	-	-	-	-	-	-	-	2	1	-
Strong musty-moldy	-	-	-	-	-	-	-	-	-	1	-	-
V. sl. putrid	-	1	-	-	-	-	-	-	-	-	-	-
Off color	-	-	-	-	-	-	1(1)	-	-	-	-	-

Total Number Judgments - 8 for each control (K) and 16 for each pouch variable.

- (1) Odor comments as noted by one person at the time of package opening.  
 (2) Inside of pouch mottled.  
 (#3) = 50M-50F-.003" Marlex 6050  
 (#4) = 50M-50F-.002" Nylon 11  
 (#8) = 50M-50F-.002" Vitel 409

that was cut and sliced to 6/64" thickness. The original slices were cut in half and both ends of the same slice were included in the package. Small bacon pieces were added to give a minimum pouch weight of 4 ounces.

12.61 Physical Packaging Tests (1 week, 3, 6, and 12 months' storage)

Weight Check

All sample packs were checked for weight change in accordance with Section 12.3, Package Evaluation Procedure. The results of the weight checks are reported in Table 51 and show 0.1% or less weight change on all test samples. This weight change is considered to be insignificant to affect product quality on these test samples.

Head Space Gas

The bacon packages were analyzed for head space gas by using Continental Can Company Test Method STM-170 (Appendix J). All packages from these inspection periods were checked by this method. The total gas volume was checked on "contingency" bacon package samples initially by using Continental Can Test Method STM-180 (Appendix K). However, since insufficient samples were available, additional total volume checks were not made by this method at the 3, 6, and 12 months' inspection period.

Observations indicated that the gas volumes appeared to remain constant in the pouches throughout the 12 months' storage period. The percent oxygen in the headspace is reported in Table 53 and shows that the oxygen content gradually increased through the 12 months'

TABLE 51

IRRADIATED POUCH TEST PACK

Product Weight Loss - (Bacon Pouches)

<u>Structure - Storage</u>	<u>Percent Product Weight Loss</u>			
	<u>Average</u>	<u>Average</u>	<u>Average</u>	<u>Range</u>
<u>77°F, 50% R.H.</u>				
50M-50F-.003" Marlex 6050	< 0.1	< 0.1	0.07	(0.07-0.08)
50M-50F-.002" Nylon 11	< 0.1	0.1	0.08	(0.07-0.08)
50M-50F-.002" Vitel 409	< 0.1	< 0.1	0.06	(0.05-0.07)
<u>100°F, 10% R.H.</u>				
50M-50F-.003" Marlex 6050	< 0.1	< 0.1	0.04	(0.03-0.05)
50M-50F-.002" Nylon 11	< 0.1	< 0.1	0.08	(0.07-0.08)
50M-50F-.002" Vitel 409	< 0.1	< 0.1	0.03	(0.03-0.05)
<u>100°F, 90% P.H.</u>				
50M-50F-.003" Marlex 6050	< 0.1	< 0.1	0.08	(0.08-0.08)
50M-50F-.002" Nylon 11	< 0.1	< 0.1	0.13	(0.03-0.18)
50M-50F-.002" Vitel 409	< 0.1	< 0.1	0.06	(0.05-0.06)

TABLE 52

## IRRADIATED POUCH TEST PACK

## Head Space Gas Analysis - (Canned Bacon Controls)

## Head Space Factors

	1 Week		3 Months		6 Months		12 Months	
GAS								
Sample No.	<u>1</u>	<u>2</u>	<u>1</u>	<u>2</u>	<u>1</u>	<u>2</u>	<u>1</u>	<u>2</u>
<u>77°F, 50% R.H.</u>								
CO <sub>2</sub>	9.9	7.9	8.8	8.8	9.1	9.3	12.0	14.3
H <sub>2</sub>	37.4	37.2	40.2	41.1	42.6	36.7	40.3	38.1
O <sub>2</sub> + A	0.65	0.67	0.7	0.7	0.7	0.5	0.5	0.6
N <sub>2</sub>	49.4	51.6	47.8	46.6	45.0	50.9	45.5	45.0
CO	2.4	2.33	2.3	2.5	2.3	2.4	1.7	2.0
CH <sub>4</sub>	0.25	0.3	0.2	0.3	0.3	0.2	-	-
Total Per Cent	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Total Headspace Volume (ml)	184.9	133.9	99.2	92.8	76.6	83.8	108.0	105.5
Vacuum in Can on Opening (inches)	9	3	5	6	4	5	5	5
<u>100°F, 10% R.H.</u>								
CO <sub>2</sub>	7.4	7.5	10.3	10.8	15.8	17.9	14.9	15.8
H <sub>2</sub>	40.0	20.0	33.5	37.1	33.9	26.0	27.8	35.4
O <sub>2</sub> + A	0.59	0.86	0.8	0.7	0.6	0.5	0.7	0.6
N <sub>2</sub>	49.66	68.9	50.0	48.9	47.3	52.8	53.0	46.3
CO	2.35	2.5	2.1	2.3	2.2	2.5	1.6	1.9
CH <sub>4</sub>	<0.1	0.24	0.3	0.2	0.2	0.3	-	-
Total Per Cent	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Total Headspace Volume (ml)	17.1	175.1	29.2	92.8	111.2	117.3	58.5	118.5
Vacuum in Can on Opening (inches)	0	8	7	5	7	7	1.5(1)	5

(1) This figure represents pressure expressed in p.s.i.

TABLE 53

IRRADIATED POUCH TEST PACK

Head Space Oxygen - (Bacon Pouches)					
<u>Storage and Structure</u>	<u>Initial Total Head Space Volume (ml)</u>	<u>1 Week</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
<u>77°F., 50% R.H.</u>					
50M-50F-.003' Marlex 6050	Avg. Range 14	0.42 (0.27-0.60)	1.22 (1.15-1.26)	1.11 (1.05-1.15)	1.43 (1.27-1.72)
50M-50F-.002' Nylon 11	Avg. Range 13	0.44 (0.27-0.72)	1.18 (1.18-1.19)	1.09 (1.07-1.12)	1.66 (1.43-1.90)
50M-50F-.002' Vitel 409	Avg. Range 12	0.61 (0.26-1.20)	1.19 (1.16-1.22)	1.09 (0.92-1.20)	2.67 (1.42-4.18)
<u>100°F., 10% R.H.</u>					
50M-50F-.003' Marlex 6050	Avg. Range 14	0.44 (0.38-0.57)	1.16 (1.09-1.20)	1.03 (0.97-1.09)	1.49 (1.34-1.49)
50M-50F-.002' Nylon 11	Avg. Range 13	0.27 (0.24-0.31)	1.13 (1.11-1.18)	1.11 (1.00-1.22)	2.10 (1.26-3.25)
50M-50F-.002' Vitel 409	Avg. Range 12	0.29 (0.27-0.31)	1.14 (1.09-1.20)	1.12 (1.10-1.14)	2.34 (1.45-3.16)
<u>100°F., 90% R.H.</u>					
50M-50F-.003' Marlex 6050	Avg. Range 14	0.61 (0.36-1.11)	1.10 (1.05-1.19)	1.11 (1.10-1.12)	1.37 (1.28-1.56)
50M-50F-.002' Nylon 11	Avg. Range 13	0.40 (0.29-0.50)	1.12 (1.09-1.19)	1.31 (1.02-1.89)	3.03 (1.44-6.54)
50M-50F-.002' Vitel 409	Avg. Range 12	0.66 (0.29-1.22)	1.06 (1.02-1.09)	1.11 (1.06-1.20)	2.01 (1.44-2.59)

storage period, being in general in the 1% to 3% at the end of the 1 year storage period. These levels are approaching sufficiently high levels to possibly cause some slight changes in organoleptic nature of the product, thus the oxygen permeability headspace content should be reviewed in terms of the organoleptic evaluation of the bacon samples to see if this oxygen increase has a gross effect on the organoleptic problems with the packaged product. The headspace gas analysis of the canned controls are reported in Table 52. The vacuum in the can controls was constant through the 12 months' storage period, as well as the oxygen content which was reported as oxygen and argon. The oxygen content appeared to reduce to essentially 0% in the can controls after the initial storage and remained this way through the 1 year storage life. A single can control stored at 100°F, 10% RH at the 1 year inspection period showed a 1-1/2 psi pressure. Since the headspace analysis compared to a can with a 5" vacuum stored under similar conditions, it was concluded that the reason for the slight pressure was due to a low vacuum at the time of closure. There appeared to be some slight fluctuation in the headspace gases in the can controls. The carbon dioxide (CO<sub>2</sub>) appeared to increase after 1 week of storage and then remained fairly constant throughout the rest of the 12 months' storage period being at the 100°F, 10% RH level. The 77°F, 50% RH storage test indicated the CO<sub>2</sub> content to be the highest only at the 12 months' storage period. However, it is believed that this is due to the adding together of the CO and CO<sub>2</sub> on the headspace analysis of the cans at 77°F, 50% RH at the 12 months' level, rather than the actual change in the CO<sub>2</sub> at this condition.

### Bond Strengths

The bond strengths were checked in the seal areas and body areas between the foil and food contacting films, by using Continental Can Company's Test Method STM-8 (Appendix A). The results of the bond strengths determinations are reported in Tables 54, 55, and 56. The heat sealing resulted in an initial increase in bond strength in the seal areas; however, this particular increase was lost during the first 3 months' storage. The bond in the seal areas and body areas were essentially the same on all test samples through the 3, 6, and 12 months' inspection periods. The bond strengths at 77°F, 50% RH appeared to be sufficient to be functional for all of these packages after the 12 months' inspection period, with there being definite lowering of the bond strength in the body areas on the 50M-50F-.003" Marlex 6050 at the 77°F, 50% RH storage. The 50M-50F-.003" Marlex 6050 maintained bond satisfactorily through the 6 months' storage at the 100°F, 10% RH and 100°F, 90% RH, but suffered total bond loss at the 12 months' storage at these 100°F storage conditions. The 50M-50F-.002" Nylon 11, likewise, showed bond strength reduction at the 12 months' inspection period at the 100°F, 10% RH, and 100°F, 90% RH storage conditions. However, the bonds in the seal area were maintained at a normal level through the 12 months' storage with this 50M-50F-.002" Nylon 11 structure. The 50M-50F-.002" Vitel 409 showed film brittleness at the 100°F, 10% RH and 100°F, 90% RH, with the film too brittle to obtain bond determination. However, these values

TABLE 54

IRRADIATED POUCH TEST PACK

Body Area Bond Strengths - (77° F., 50% R.H. - Bacon Pouches)

Structure - Body Area (1)	Initial (2) *	Grams/Linear Inch Width (3)			12 Months *
		1 Week *	3 Months *	6 Months *	
<u>50M-50F-.003' Marlex 6050</u>	Avg. Range 910 (850-950)				
Right Side Area	Avg. Range	560 (380-900)	400 (320-550)	430 (330-470)	310 (200-350)
Left Side Area	Avg. Range	510 (320-960)	400 (350-500)	420 (400-450)	320 (200-400)
Bottom Area	Avg. Range	500 (380-710)	360 (310-430)	410 (350-450)	70 (0-300)
<u>50M-50F-.002' Nylon 11</u>	Avg. Range 560 (560-560)				
Right Side Area	Avg. Range	470 (320-500)	420 (350-450)	370 (350-420)	310 (300-350)
Left Side Area	Avg. Range	380 (300-520)	470 (250-550)	390 (350-450)	330 (300-400)
Bottom Area	Avg. Range	400 (320-480)	460 (320-550)	370 (350-400)	280 (350-400)
<u>50M-50F-.002' Vitel 409</u>	Avg. Range 593 (510-650)				
Right Side Area	Avg. Range	590 (470-680)	430 (370-470)	510 (450-600)	590 (500-600)
Left Side Area	Avg. Range	560 (470-700)	430 (320-500)	520 (400-650)	610 (500-650)
Bottom Area	Avg. Range	470 (400-500)	410 (350-450)	520 (500-580)	560 (500-650)

(1) Body areas between the foil and food contacting film.

(2) Bonds checked on stock at the time pouches were manufactured.

(3) Continental Can Company Test Method STM-8 (Appendix A).

TABLE 55

## IRRADIATED POUCH TEST PACK

Body Area Bond Strengths - (100° F., 10% R.H. - Bacon Pouches)

Structure - Body Area (1)	Initial (2) *	Grams/Linear Inch Width (3)			12 Months *
		1 Week *	3 Months *	6 Months *	
50M-50F-.003' Marlex 6050	Avg. Range 910 (850-950)				
Right Side Area	Avg. Range	460 (320-800)	300 (300-300)	450 (450-450)	0 (0-0)
Left Side Area	Avg. Range	440 (350-650)	340 (280-380)	400 (350-450)	0 (0-0)
Bottom Area	Avg. Range	490 (410-730)	340 (320-350)	420 (350-450)	0 (0-0)
50N-50F-.002' Nylon 11	Avg. Range 560 (560-560)				
Right Side Area	Avg. Range	370 (300-450)	310 (280-340)	370 (350-450)	200 (150-250)
Left Side Area	Avg. Range	430 (300-580)	310 (280-340)	390 (350-500)	150 (0-200)
Bottom Area	Avg. Range	350 (300-450)	300 (280-320)	370 (350-400)	180 (0-250)
50M-50F-.002' Vitel 409	Avg. Range 593 (510-650)				
Right Side Area	Avg. Range	450 (410-480)	410 (350-450)	470 (400-550)	330 (300-380)
Left Side Area	Avg. Range	450 (400-500)	440 (380-480)	480 (350-600)	320 (250-350)
Bottom Area	Avg. Range	440 (420-450)	440 (400-470)	490 (400-550)	260 (0-450)

- (1) Body areas between the foil and food contacting film  
 (2) Bonds checked on stock at the time pouches were manufactured.  
 (3) Continental Can Company Test Method STN-8 (Appendix A).

TABLE 56

IRRADIATED POUCH TEST PACK

Body Area Bond Strengths - (100°F., 90% R.H. - Bacon Pouches)

Structure - Body Area (1)	Initial (2) *	Grams/Linear Inch Width (3)			
		1 Week *	3 Months *	6 Months *	12 Months *
<u>50M-50F-.003' Marlex 6050</u>	Avg. Range	910 (850-950)			
Right Side Area	Avg. Range	460 (350-600)	280 (180-380)	420 (350-450)	0 (0-0)
Left Side Area	Avg. Range	390 (330-600)	330 (250-440)	420 (400-480)	0 (0-0)
Bottom Area	Avg. Range	370 (350-390)	300 (220-380)	420 (350-450)	0 (0-0)
<u>50M-50F-.002' Nylon 11</u>	Avg. Range	560 (560-560)			
Right Side Area	Avg. Range	390 (230-490)	330 (260-480)	270 (250-350)	130 ( 50-200)
Left Side Area	Avg. Range	420 (320-500)	340 (280-450)	350 (200-480)	55 ( 30- 80)
Bottom Area	Avg. Range	340 (310-370)	350 (300-450)	320 (200-450)	170 ( 80-300)
<u>50M-50F-.002' Vitel 409</u>	Avg. Range	593 (510-650)			
Right Side Area	Avg. Range	520 (450-620)	480 (450-510)	420 (350-500)	***
Left Side Area	Avg. Range	520 (450-700)	500 (420-570)	440 (350-500)	***
Bottom Area	Avg. Range	440 (400-520)	450 (420-470)	410 (350-450)	***

- (1) Body areas between the foil and food contacting film.  
 (2) Bonds checked on stock at the time pouches were manufactured.  
 (3) Continental Can Company Test Method STM-8 (Appendix A).

TABLE 57

IRRADIATED POUCH TEST PACKSeal Area Bond Strength - (77°F, 50% R.H. - Bacon Pouches)

<u>Structure - Seal Areas (2)</u>		<u>Grams/Linear Inch Width (1)</u>			
		<u>1 Week</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
		*	*	*	*
<u>50M-50F-.003" Marlex 6050</u>					
Right Seal Area	Average	700	470	380	380
	Range	(480-800 )	(400-600)	(350-400)	(350-400)
Left Seal Area	Average	820	470	360	380
	Range	(740-900 )	(420-550)	(350-370)	(300-430)
Bottom Seal Area	Average	660	460	340	325
	Range	(600-800 )	(400-500)	(300-400)	(300-350)
<u>50M-50F-.002" Nylon 11</u>					
Right Seal Area	Average	940	510	320	360
	Range	(750-1000+)	(450-550)	(250-400)	(350-400)
Left Seal Area	Average	980	530	360	350
	Range	(830-1000+)	(400-600)	(310-450)	(300-350)
Bottom Seal Area	Average	980	540	380	400
	Range	(900-1000+)	(400-650)	(280-500)	(350-450)
<u>50M-50F-.002" Vitel 409</u>					
Right Seal Area	Average	740	520	470	640
	Range	(700-800 )	(450-650)	(400-550)	(600-650)
Left Seal Area	Average	740	500	490	610
	Range	(440-870 )	(430-620)	(350-600)	(600-650)
Bottom Seal Area	Average	710	480	480	600
	Range	(630-820 )	(450-500)	(450-530)	(550-650)

(1) Continental Can Company Test Method STM-6 (Appendix A).

(2) Seal areas between the foil and food contacting film.

TABLE 58

IRRADIATED POUCH TEST PACKSeal Area Bond Strength - (100°F, 10% R.H. - Bacon Pouches)

<u>Structure - Seal Area (3)</u>		<u>Grams/Linear Inch Width (1)</u>			
		<u>1 Week</u> *	<u>3 Months</u> *	<u>6 Months</u> *	<u>12 Months</u> *
<u>50M-50F-.003" Marlex 6050</u>					
Right Seal Area	Average	890	440	370	0
	Range	(670-1000 )	(350-480)	(300-400)	( 0-0 )
Left Seal Area	Average	880	400	360	0
	Range	(610-1100 )	(350-480)	(300-400)	( 0-0 )
Bottom Seal Area	Average	760	400	390	0
	Range	(650-950 )	(400-420)	(300-450)	( 0-0 )
<u>50M-50F-.002" Nylon 11</u>					
Right Seal Area	Average	830	410	330	330
	Range	(720-1000+)	(350-480)	(300-400)	(200-300)
Left Seal Area	Average	830	400	340	250
	Range	(700-1000 )	(320-480)	(280-450)	(200-300)
Bottom Seal Area	Average	840	370	350	250
	Range	(740-980 )	(350-400)	(300-400)	(200-300)
<u>50M-50F-.002" Vitel 409</u>					
Right Seal Area	Average	790	480	450	(2)
	Range	(740-830 )	(400-500)	(350-500)	
Left Seal Area	Average	760	510	420	(2)
	Range	(600-920 )	(480-550)	(350-500)	
Bottom Seal Area	Average	650	480	450	(2)
	Range	(500-800 )	(430-500)	(400-500)	

- (1) Continental Can Company Test Method STM-8 (Appendix A).  
 (2) Vitel 409 film too brittle to permit bond determination.  
 (3) Seal areas between the foil and food contacting film.

TABLE 59

IRRADIATED POUCH TEST PACKSeal Area Bond Strength - (100°F, 90% R.H. - Bacon Pouches)

<u>Structure - Seal Area (3)</u>		<u>Grams/Linear Inch Width (1)</u>			
		<u>1 Week</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
		*	*	*	*
<u>50M-50F-.003" Marlex 6050</u>					
Right Seal Area	Average	770	430	370	0
	Range	(750-780 )	(400-450)	(300-400)	( 0-0 )
Left Seal Area	Average	790	460	400	0
	Range	(720-810 )	(440-500)	(350-450)	( 0-0 )
Bottom Seal Area	Average	660	410	370	0
	Range	(500-750 )	(340-480)	(250-450)	( 0-0 )
<u>50M-50F-.002" Nylon 11</u>					
Right Seal Area	Average	900	430	240	340
	Range	(770-1000+)	(320-550)	(180-300)	(280-350)
Left Seal Area	Average	890	430	320	340
	Range	(770-1000+)	(350-580)	(200-480)	(280-380)
Bottom Seal Area	Average	940	420	280	310
	Range	(860-1000+)	(350-500)	(180-400)	(250-400)
<u>50M-50F-.002" Vitel 409</u>					
Right Seal Area	Average	790	580	360	(2)
	Range	(720-850 )	(550-620)	(300-400)	
Left Seal Area	Average	730	570	390	(2)
	Range	(440-870 )	(520-650)	(300-450)	
Bottom Seal Area	Average	800	540	370	(2)
	Range	(680-860 )	(500-600)	(300-400)	

- (1) Continental Can Company Test Method STM-8 (Appendix A).  
 (2) Vitel 409 film too brittle to permit bond determination.  
 (3) Seal areas between the foil and food contacting film.

were probably in the 300 gram range. Thus, only the 50M-50F-.002" Nylon 11 laminate appeared to offer satisfactory bond performance through the total 12 months' storage conditions under all exposure conditions. The bacon fat apparently degraded the film on the 50M-50F-.002" Vitel 409 and permeated the 50M-50F-.003" Marlex 6050 sufficiently to cause bond loss at the 100°F storage conditions.

#### Seal Strengths

The seal strengths were checked on the side seals and bottom seals (both stock directions) by using Continental Can Company Test Method STM-13 (Appendix B). The results of the seal strength tests are reported in Tables 60 through 62. The seal strengths were maintained on the 50M-50F-.002" Nylon 11 laminate under all test conditions. The 50M-50F-.003" Marlex 6050 laminate suffered seal strength lowering in the bottom seal area which is the machine direction of the stock indicating fat from the product was affecting the linear polymeric structure. This lowering of seal strength in the body seal area is sufficient to be in the critical range for adequate package durability. The 50M-50F-.002" Vitel 409 showed fairly consistent maintenance of seal strengths at 77°F, 50% RH through the six months' storage period; however, after 12 months' storage at this condition, considerable bond loss was noted. The 50M-50F-.002" Vitel 409 showed a gradual loss of seal strength at 100°F, 90% RH and 100°F, 10% RH with the seal strengths being totally inadequate at the 12 month storage period. This was due to film embrittlement with

TABLE 60

IRRADIATED POUCH TEST PACK

Seal Strengths - (77° F., 50% R.H. - Bacon Pouches)

Structure-Seal Area	Initial (2) *	Grams/Linear Inch Width (1)		
		1 Week *	3 Months *	6 Months * 12 Months *
<u>50M-50F-.003"Marlex 6050</u>				
Right Side Seal	Avg. Range 5700 (5300-6000)	4800 (3900-5500)	4300 (2200-5600)	4800 (3000-5800) 4600 (4500-6000)
Left Side Seal	Avg. Range 5700 (5300-6000)	4700 (4400-5200)	4500 (4100-5100)	4700 (4000-5400) 4600 (2800-5400)
Bottom Seal	Avg. Range 5000 (4800-5400)	1500 (1000-1900)	2100 (1000-4800)	1600 ( 800-3800) 1300 ( 850-1500)
<u>50M-50F-.002'Nylon 11</u>				
Right Side Seal	Avg. Range 5300 (5000-5600)	5100 (4400-5500)	4400 (3800-4900)	4800 (3900-5200) 4500 (3800-5300)
Left Side Seal	Avg. Range 5300 (5000-5600)	5100 (4900-5600)	4300 (3900-4800)	4600 (4500-4800) 4800 (4500-5000)
Bottom Seal	Avg. Range 5100 (4600-5300)	4000 (3400-4500)	4100 (3100-4500)	3900 (2300-4800) 4600 (4500-5200)
<u>50M-50F-.002"Vitel 409</u>				
Right Side Seal	Avg. Range 4400 (3900-4900)	4300 (3200-4600)	4400 (3500-5200)	4300 (3400-4800) 2100 ( 800-4100)
Left Side Seal	Avg. Range 4400 (3900-4900)	4300 (3900-4600)	4500 (3800-5400)	4100 (3900-4200) 1600 ( 800-4100)
Bottom Seal	Avg. Range 4200 (3100-4900)	4300 (3700-5000)	3600 ( 800-5000)	4200 (2900-4800) 3100 (2500-4100)

(1) Continental Can Company Test Method STM-13 (Appendix B).

(2) Seals checked on stock at the time pouches were manufactured.

TABLE 61

IRRADIATED POUCH TEST PACK

Seal Strengths - (100°F., 10% R.H. - Bacon Pouches)

<u>Structure-Seal Area</u>	<u>Initial (2)</u> *	<u>Grams/Linear Inch Width (1)</u>		
		<u>1 Week</u> *	<u>3 Months</u> *	<u>6 Months</u> *
				<u>12 Months</u> *
<u>50M-50F-.003"Marlex 6050</u>				
Right Side Seal	Avg. Range 5700 (5300-6000)	5100 (4100-5400)	4800 (4000-5400)	4300 (1600-5400)
Left Side Seal	Avg. Range 5700 (5300-6000)	4700 (3700-5200)	5100 (5000-5200)	4800 (3100-5700)
Bottom Seal	Avg. Range 5000 (4800-5400)	1100 ( 900-1200)	3300 ( 900-5200)	1900 (1100-3100)
<u>50M-50F-.002"Nylon 11</u>				
Right Side Seal	Avg. Range 5300 (5000-5600)	4800 (4400-5400)	4700 (3700-5400)	4400 (3400-5000)
Left Side Seal	Avg. Range 5300 (5000-5600)	4700 (4300-5200)	4300 (3600-4800)	4400 (3900-5000)
Bottom Seal	Avg. Range 5100 (4600-5300)	3300 (2700-4000)	4100 (3600-4800)	4200 (3500-4800)
<u>50M-50F-.002"Vitel 409</u>				
Right Side Seal	Avg. Range 4400 (3900-4900)	3900 (2800-4600)	2100 ( 800-4000)	2700 (1800-4000)
Left Side Seal	Avg. Range 4400 (3900-4900)	4000 (3200-4600)	2700 ( 800-4500)	2700 (1500-3900)
Bottom Seal	Avg. Range 4200 (3100-4900)	4300 (4200-4600)	3100 (2000-4700)	3000 (1800-3700)

- (1) Continental Can Company Test Method STM-13 (Appendix B).  
 (2) Seals checked on stock at the time pouches were manufactured.

TABLE 62

IRRADIATED POUCH TEST PACK

Seal Strengths - (100°F., 90% R.H. - Bacop Pouches)

Structure-Seal Area	Initial (2) *	Grams/Linear Inch Width (1)			
		1 Week *	3 Months *	6 Months *	12 Months *
<u>50M-50F-.003" Marlex 6050</u>					
Right Side Seal	Avg. Range	5700 (5300-6000†)	4700 (3400-5300)	4900 (3600-5800)	4800 (3500-6000)  4200 (2400-4800)
Left Side Seal	Avg. Range	5700 (5300-6000†)	5000 (4700-5300)	5000 (4800-5100)	4900 (4000-5900)  3300 (2800-3700)
Bottom Seal	Avg. Range	5000 (4800-5400)	1900 (1300-2700)	2000 ( 900-4200)	2100 (1000-5800)  3800 (2700-3800)
<u>50M-50F-.002" Nylon 11</u>					
Right Side Seal	Avg. Range	5300 (5000-5600)	4900 (4100-5400)	4200 (3500-4600)	4400 (3800-4900)  4600 (3600-5200)
Left Side Seal	Avg. Range	5300 (5000-5600)	4700 (4100-5000)	4000 (3400-4400)	4400 (4200-4700)  4800 (4600-5100)
Bottom Seal	Avg. Range	5100 (4600-5300)	3800 (2600-5000)	3900 (2400-4800)	4200 (3800-4400)  4200 (3900-4600)
<u>50K-50F-.002" Vitel 409</u>					
Right Side Seal	Avg. Range	4400 (3900-4900)	4100 (2200-4600)	1000 ( 700-1500)	1200 (1100-1300)  750 ( 300-1300)
Left Side Seal	Avg. Range	4400 (3900-4900)	3800 (3000-4100)	1000 ( 700-1300)	1400 (1300-1500)  1500 ( 600-3500)
Bottom Seal	Avg. Range	4200 (3100-4900)	4500 (4300-5400)	2700 (1900-3500)	2200 (1800-2900)  600 ( 500-700 )

(1) Continental Can Company Test Method STM-13 (Appendix B).

(2) Seals checked on stock at the time pouches were manufactured.

this high fat product being more severe at the 100°F storage conditions. Thus only the 50M-50F-.002" Nylon 11 was satisfactory on seal strengths under all exposure conditions.

#### Observations

There was no noted delamination or other package change observed other than the above reported embrittlement of the Vital 409 film and the reported bond and seal strength variations.

#### 12.62 Organoleptic Testing (1 week, 3, 6, and 12 months' storage)

##### Sensory Difference Tests

The flavor of the irradiated bacon in the three pouch variables was determined to be "very slightly different" from that of the irradiated bacon in the can controls. The range of difference scores for the three package types was 0.10 to 1.22. No real difference in the organoleptic performance of the three pouch types was indicated. A higher percentage of difference judgments was obtained with pouch 50M-50F-.003" Marlex 6050 than was obtained in the other two pouches. This difference was consistent with a trend noted in the irradiated ham and chicken studies (See Table 64).

Sensory difference panelists described the bacon odor as "burnt." Rancid odor was reported more frequently in the can control than in the pouch samples (See Table 65). The bacon flavor was described as "burnt" with "rancid" flavor occurring in both pouch and can control

samples (See Table 66). The overall quality of the irradiated bacon from the metal can controls was judged to be "good-to-fair" on a scale of "excellent," "good," "fair," or "poor" quality (See Table 63).

#### "In-Package" Odor Tests

Odor intensity scores recorded at the time of package opening by one person ranged from "0" or "none" to "2.75" or "moderate" for the bacon pouch variables. The can control intensity scores ranged from "0" or "no odor" to "1.00" or "trace odor" (See Table 67).

Comments recorded during the "in-package" sniff tests indicated that the character of the bacon odor was "smoky" at 75°F, 50% RH storage conditions and "burnt" or "burnt-smoky" at the higher temperature storage conditions at 1, 3, and 6 months' storage. After 12 months' storage, however, the bacon odor was described as "burnt" under all conditions (See Table 68).

### 13. STUDY OF INSECT PENETRATION INTO FLEXIBLE PACKAGES\*

#### 13.1 Objective

To evaluate the resistance to penetration of three types of irradiated flexible packages to three insect species.

#### 13.2 Method

The following material variables were included in this test:

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\* Work performed under Contract to CCC by the Wisconsin Alumni Research Foundation, Pesticide Department; Mr. Gilbert Schmolesky was in charge of the project.

Irradiated Samples

Non-Irradiated Samples

1-50M-50F-.003" Marlex 6050	1-50M-50F-.003" Marlex 6050
2-50M-50F-.002" Nylon 11	2-50M-50F-.002" Nylon 11
3-50M-50F-.002" Vitel 409	3-50M-50F-.002" Vitel 409
	4-50M-.003" Vitel 409

The 50M-.003" Vitel 409 pouches were provided to include a non-foil structure in this test for comparison purposes. The irradiated samples were obtained from the test pack samples and included ham which was irradiated at 4.5 megarads. A similar type of ham was obtained for the non-irradiated samples. The ham was packed in the same pouch and product size, processed at 250°F for 30 minutes in a thermo retort in order to prevent product spoilage during the insect penetration period. All of these test materials were glued in the protective jackets and packed in the inner corrugated cartons as was done in the case of the test pack. In order to determine the effect of pouch durability and insect penetration, one set of samples was supplied without exposure to an abuse treatment and the other set of samples was supplied with a shipping exposure of lg. for 1 hour by method ASTM-D99-63T. All package samples were tested in quadruplicate for each of the processing and handling variables.

Test Insects

Three species of insects known to have ability to penetrate food packages were used in this evaluation.

TABLE 63

IRRADIATED POUCH TEST PACK

ORGANOLEPTIC TESTS

AVERAGE "QUALITY" SCORES FOR BACON IN METAL CANS  
AT VARIOUS TIME PERIODS (1)

IRRADIATED METAL CAN CONTROL

SCALE

Excellent = 1

Good = 2

Fair = 3

Poor = 4

<u>Storage Conditions</u>	<u>1 Month</u>	<u>3 Months</u>	<u>6 Months</u>	<u>12 Months</u>
75°F - 50% R.H.	2.6	2.3	3.1	2.6
100°F - 10% R.H.	2.7	2.5	2.9	2.8
100°F - 90% R.H.	2.4	2.5	3.5	2.5

(1) Evaluated by paneling prior to judging "Difference."

TABLE 64

IRRADIATED POUCH TEST PACKS  
ORGANOLEPTIC TESTS

Flavor difference scores of (1) bacon pouches vs metal cans

Pouch Types	77° F. - 50% R.H.				100° F. - 10% R.H.				100° F. - 90% R.H.			
	1 mo.	3 mo.	6 mo.	12 mo.	1 mo.	3 mo.	6 mo.	12 mo.	1 mo.	3 mo.	6 mo.	12 mo.
50M-50F-.003"Marlex 6050	0.22	0.78	1.14	0.80	0.38	0.44	0.86	0.30	1.22	0.67	0.71	0.40
50M-50F-.002" Nylon 11	0.78	0.67	0.29	0.50	0.13	0.11	1.29	0.20	0.33	0.33	0.71	0.30
50M-50F-.002" Vitel 409	0.67	0.89	0.57	0.80	0.75	0.33	1.29	0.40	0.89	0.56	0.14	0.10

Flavor difference scores of (1) Bacon pouches vs. metal cans

Storage Conditions	50M-50F-.003"Marlex 6050	50M-50F-.002"Nylon 11	50M-50F-.002"Vitel 409
77° F. - 50% R.H.	0.71	0.57	0.74
100° F. - 10% R.H.	0.47	0.38	0.65
100° F. - 90% R.H.	0.74	0.29	0.43
	Avg. 0.64	Avg. 0.45	Avg. 0.61

PER CENT OF BACON PANEL FINDING A "FLAVOR DIFFERENCE" (2)

Pouch Structure	50M-50F-.003"Marlex 6050	50M-50F-.002" Nylon 11	50M-50F-.002" Vitel 409
	40.4%	27.9%	38.5%

(1) Difference of pouch sample from metal can control as scored on a 0-5 Scale

(2) Total number of judgments: 312

TABLE 65

IRRADIATED POUCH TEST PACK

ORGANOLEPTIC TESTS

SUMMARY OF IRRADIATED BACON "ODOR" COMMENTS (1)

<u>Odor Comments</u>	<u>#3</u>	<u>#4</u>	<u>#8</u>	<u>K</u>
Irradiated Odor:	2	1	-	-
Scorched	-	-	-	3
Smoked-smoky	1	-	1	1
Burnt	10	6	8	18
Cooked-smoky				
None				
V. Slight	2	-	1	4
Slight	-	-	-	3
Moderate	9	4	7	8
Strong	-	-	-	7
Less (than K)	1	2	1	-
More (than K)	1	1	-	-
Pungent (tar-like)				
Slight	-	-	1	-
Less (than K)	1	-	-	-
Bland-odorless	1	1	-	2
Appropriate (typical)	3	-	2	15
Non-appropriate (disagreeable)	-	-	-	4
Off (other than irradiated)	3	1	1	-
Sour	-	-	-	1
Musty	1	-	-	-
Stale	2	-	-	1
Rancid	-	-	1	6
Salty				
TOTAL NUMBER JUDGMENTS	37	37	37	37

- (1) Bacon "odor" comments as made during the sensory difference test.  
 (#3) = 50M-50F-.003" Marlex 6050  
 (#4) = 50M-50F-.002" Nylon 11  
 (#8) = 50M-50F-.002" Vitel 409  
 K = Can control

TABLE 66  
IRRADIATED POUCH TEST PACK  
ORGANOLEPTIC TESTS

SUMMARY OF IRRADIATED BACON "FLAVOR" COMMENTS\*

<u>Odor Comments</u>	<u>#3</u>	<u>#4</u>	<u>#8</u>	<u>Can</u>
Irradiated Flavor:	2	2	1	1
Scorched	-	-	1	2
Smoked-smoky	-	-	1	2
Smoked-fishy	-	-	1	2
Smoked-burnt	-	-	1	2
Burnt	5	7	9	5
Cooked	-	-	1	-
Cooked-smoky	-	-	1	-
None	-	-	1	-
V. Slight	-	1	1	1
Slight	2	2	2	-
Moderate	4	3	3	5
Strong	-	-	-	-
Less (than K)	1	1	1	-
More (than K)	2	2	4	-
Pungent (tar-like)	-	-	-	-
Slight	-	1	-	-
Less	-	-	-	-
More	-	-	-	-
Weaker than odor	-	-	-	-
Bland-tasteless	1	1	2	6
Appropriate (typical)	3	3	1	4
None	-	-	-	-
V. Slight	-	-	-	-
Non-appropriate (disagreeable)	-	-	-	2
Off (other than irradiated)	2	-	1	-
None	1	1	2	-
Strong-bitter	-	-	-	1
Cardboard	1	-	-	-
Stale	-	-	-	2
Rancid	3	2	1	2
Metallic	-	1	-	-
Oxidized	-	1	1	1
Salty	1	-	4	2
Spicy	-	-	-	-
Sweet	-	-	3	1
Overcooked	1	1	1	-
Undercooked (fatty)	2	-	-	4
Packing house	-	-	-	-
Better than K	-	-	-	-
Drier	-	-	-	-
Tough	-	-	-	-
Moist	-	-	-	-
Soft-mushy	-	-	-	2
Greasy	-	-	2	2
TOTAL NUMBER JUDGMENTS	37	37	37	37

\* Bacon "flavor" comments as made during the sensory difference test.  
 (#3) = 50M-50F-.003" Marlex 6050  
 (#4) = 50M-50F-.002" Nylon 11  
 (#8) = 50M-50F-.002" Vitel 409

TABLE 67

IRRADIATED POUCH TEST PACKORGANOLEPTIC TESTSSUMMARY OF ODOR INTENSITY SCORES (1)BACON "IN-PACKAGE" ODOR TEST

0 = No Off Odor

1 = Trace Off Odor

2 = Slight Off Odor

3 = Moderate Off Odor

4 = Strong Off Odor

5 = Extremely Strong Off Odor

<u>Storage Conditions</u>			<u>Can Control</u>	(a)	(b)	(c)	<u>Average (Pouches Only)</u>
<u>Product</u>	<u>°F/% Humidity</u>	<u>Months</u>		<u>Pouch 3</u>	<u>Pouch 4</u>	<u>Pouch 8</u>	
Bacon	75/50	1	0	2.75	2.50	2.25	2.50
		3	0	1.75	1.00	1.75	1.50
		6	0	0.75	1.25	1.00	1.00
		12	0	1.75	1.50	0.25	1.17
		<u>Average</u>	0	1.75	1.56	1.31	
	100/10	1	0.50	2.00	1.50	1.75	1.75
		3	1.00	2.00	1.00	2.00	1.67
		6	0	0.50	1.50	1.25	1.08
		12	0	0.75(2)	0.75(2)	0.25(2)	0.58
		<u>Average</u>	0	1.31	1.19	1.31	
	100/90	1	0.50	1.25	1.75	2.00	1.67
		3	1.00	2.50	1.75	2.75	2.00
		6	0	1.00	0.75	1.75	1.17
		12	0	0.25	0.50	0	0.25
		<u>Average</u>	0	1.25	1.19	1.63	

(1) Each pouch score represents an average of 4 judgments made by one person; each control score represents an average of 2 judgments.

(2) Pouch material layers were separated.

(a) 50M-50F-.003" Marlex 6050

(b) 50M-50F-.002" Nylon 11

(c) 50M-50F-.002" Vital 409

TABLE 68

IRRADIATED POUCH TEST PACK

ORGANOLEPTIC TESTS

SUMMARY OF ODOR COMMENTS (1)

BACON "IN-PACKAGE" ODOR TESTS

	<u>75°F - 50% R.H.</u>				<u>100°F - 10% R.H.</u>				<u>100°F - 90% R.H.</u>			
<u>Comments:</u>	<u>K</u>	<u>3</u>	<u>4</u>	<u>8</u>	<u>K</u>	<u>3</u>	<u>4</u>	<u>8</u>	<u>K</u>	<u>3</u>	<u>4</u>	<u>8</u>
Smoky	-	12	12	12	-	-	-	-	-	8	8	8
Burnt-smoky	-	-	-	-	-	12	12	12	-	-	-	-
Burnt	-	3	2	2	-	3	3	1	-	5	6	4
Tar-like	-	11	10	9	-	11	11	9	-	5	10	8
Sl. tar-like	-	-	-	-	-	-	-	-	-	-	-	-
V. sl. tar-like	-	-	-	-	3	-	-	-	-	-	-	-
Sl. musty	-	-	-	-	-	1	-	-	-	-	-	-
Sour	-	-	-	-	-	-	-	-	-	-	1	-
V. sl. sour	1	-	-	-	-	-	1	-	-	-	-	-
V. sl. rancid	2	-	-	-	-	-	-	-	-	-	-	-
Putrid	-	-	-	-	-	-	-	-	-	-	2	-

Total Number Judgments - 8 for each control (K) and 16 for each pouch variable.

- (1) Odor comments as noted by one person at the time of package opening.  
 (#3) = 50M-50F-.003" Marlex 6050  
 (#4) = 50M-50F-.002" Nylon 11  
 (#8) = 50M-50F-.002" Vitel 409

- 1 - Trogoderma glabrum, larvae
- 2 - The lesser grain borer, Rhyzopertha dominica (F adults)
- 3 - The black carpet beetle, Attagenus piceus (Oliver)

larvae

#### Insect Penetration Test Containers

Fiberboard drums measuring 16" in diameter x 16" in height and having metal bases and covers were used to retain the test insects and pouches. These were maintained at standard temperature and humidity conditions.

#### Test Procedure

The outer jackets on each test package were folded inside out, exposing the pouches. The sample packages were placed in the holding containers on edge in random fashion. Each container held a total of 36 packages and 500 insects were placed in each. Damage observations were made every other week and all dead insects were replaced on a weekly basis (See Figure 11).

#### 13.3 Results

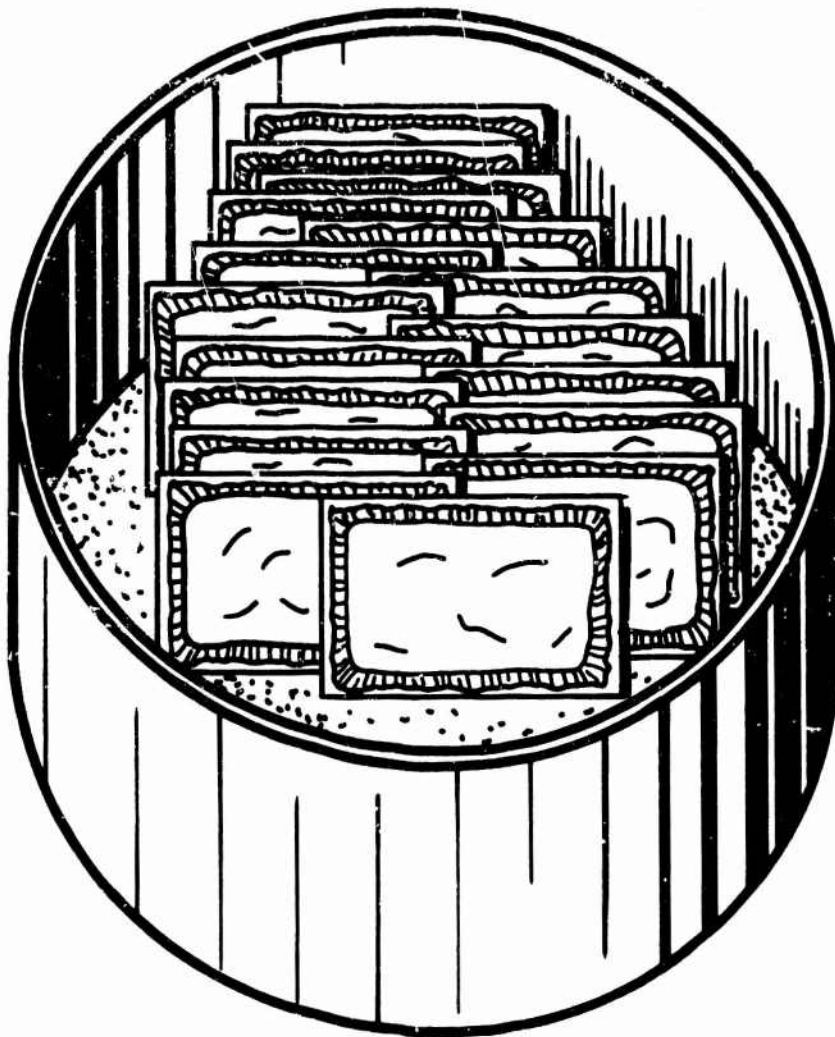
The only visible damage which occurred during the first two months was scuffing of the outer jackets. The amount of scuffing increased with time, but no drilling of the pouches or jackets occurred until 2-1/2 months. At 2-1/2 months, definite holes were drilled through jackets of two packages by the lesser grain borer. The outer foil portion of a 50M-50F-.002" Nylon 11 non-irradiated pouch was penetrated in one instance by *Trogoderma* and in another instance by a black carpet

beetle. These foil penetrations were on packages in which the foil and the .002" Nylon 11 did not have uniform bond, i.e., delamination had occurred between the foil and the Nylon causing a tube (vein) in to which the insects penetrated. The insects did not penetrate through the Nylon 11 to the food product contained in the pouch. The exposure to the insects was continued through three months and showed no complete penetration of any of the packages during the total three month storage period.

#### 13.4 Observations

The 50M-50F-.002" Nylon 11 stock was not designed for thermo processing. As a result, the material showed some delamination as the result of the thermo process exposure. This delamination resulted in insects penetrating between the foil and the Nylon 11 ply, but, did not result in penetration of the insects to the food product. The shipping and non-shipped exposed variables and irradiated and non-irradiated variables run in this test did not become significant factors in this evaluation since insect penetration was not encountered. Also, it should be pointed out that the foil used in these pouches was not necessarily a deterrent to insect penetration since the insects did not penetrate the Nylon 11 ply in the delaminated thermo processed 50M-50F-.002" Nylon 11 stock and since the insects did not penetrate the transparent 50M-.003" Vitel 409 foil pouch. Since the insects did not penetrate any pouch plies in the case of irradiated samples and did not

FIGURE II



INSECT TEST EQUIPMENT

totally penetrate any of the packages under any of the test variables, we would conclude from this test that the insect penetration resistance of these irradiated food pouches were very good and insect penetration on these pouches should not be a serious problem in usage of these pouches.

#### 14. STUDY OF FLEXIBLE PACKAGES UNDER VARIABLE TEMPERATURE CONDITIONS

##### 14.1 Objective

This study was directed towards determining if the handling of pouches at temperatures ranging from 75°F to minus 320°F, as would be encountered in irradiation exposure, would damage the pouches.

##### 14.2 Method

Pouches fabricated from the laminate materials reported in Section 11, were used for this test program. Each pouch was filled with 4 ounces of a commercial, semi-moist dog food having 25% moisture content and preserved with sugar and mold inhibitors. The headspace gas was removed from the pouches by hand manipulation and the pouches then sealed on a laboratory Sentinel Sealer, Model 12A. The pouches were then enclosed in protective jackets, with the pouch face being glued to the protective jackets, as per Figure 3 and packed in the standard shipping cases. Each case contained 42 pouches. One case of each of the test materials was prepared for condition exposure at each of the below temperature conditions of exposures:

<u>Test Materials</u>	<u>Temperature Conditions</u>
50M-50F-.003" Marlex 6050	75°F
50M-50F-.002" Nylon 11	0°F
50M-50F-.002" Vitel 409	-40°F
	-70°F
	-320°F

These prepared samples were exposed (1 carton each to each of the temperature conditions) recording the internal temperature by placing thermocouple leads in the cartons and several test pouches at various locations. When the temperature condition was reached, the samples were exposed to a shipping vibration test of (1 g.) of force for 5 minutes using method ASTM-D999-63T while maintained at these temperature conditions. Following this exposure, the pouches were conditioned at 50% RH, 77°F in storage for 2 days. Subsequently, all pouches and blanks (sealed empty pouches and jackets) were weighed and stored at 100°F, 10% RH for 30 days. At the end of the 30-day period, all pouches were brought to 50% RH, 77°F for 24 hours and weighed and the weight change noted. Any change in weight of the blanks were discarded as package material weight change.

#### 14.3 Observations

The results of this testing are reported in Table 69. The weight change of all of these samples, including the minus 320°F, were found to be negligible and the results of this testing showed no package

breakage or rupture of any of these test samples from this exposure. Since the weight change was so low at 30 days exposure, an additional 30 day exposure re-check was run on all test samples, except the minus 320°F. The results of this re-check are also reported in Table 69. The re-checks, likewise, showed negligible weight loss, thus indicating again, that the protective properties of the materials were not damaged by this abuse at these various temperature exposures. The inconsistencies of the minus 70°F initial 30 day check was found to be due to a mishandling of the blanks, in that the blank pouches were not included with the test samples at the minus 70°F exposure. However, the additional replica of the minus 70°F pouches indicate a similar performance level to the other test samples. Because the pouches were glued into the protective jackets for this exposure in order to simulate the actual package handling, the actual weight changes of the blanks were greater than the test pouches, thus causing the variation in plus and minus reported weight gains.

The results of this testing indicates that these types of flexible packages would perform satisfactorily after exposure to these conditions during the low temperature food irradiation cycle.

## 15. STUDY OF MICROBIAL PENETRATION INTO FLEXIBLE PACKAGES

### 15.1 Objective

To develop a standard, reproducible, method for evaluating the integrity of flexible packaging systems containing commercially sterile, low acid foods. More specifically, the test method was directed toward

evaluating flexible packaging systems in which the prepackaged foods were radiation sterilized at 4.5 megarad (minimum) and subjected to handling and abuse conditions simulating the level of abuse which might be encountered in military distribution and usage by individual military personnel in field conditions. The "tentative" test method should be of such a character that it could be the basis for a "standard" flexible package integrity test for military usage.

#### 15.2 Package Integrity Test Method - Premise

During the past fifteen years, a number of investigators have studied the materials and methodologies of using flexible, laminate packages for commercially sterile, low acid foods. For example, Procter and Nickerson<sup>1</sup> studied the microbial penetration through a variety of flexible films. Szczablowski and Rubinate<sup>2</sup> evaluated the performance of flexible packages, containing actual foods, by observing the type and rate of failure of flexible packages carried by military personnel during their traversing of a combat training course. Long<sup>3</sup> discussed the behavior of flexible pouches for sterile foods.

The intent of our work was to include the concepts of previously published data in establishing the criterion for the development of a standard method. In conferences held with representatives from the U. S. Army Natick Laboratories, several basic assumptions were used to

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<sup>1</sup> Procter, B. E. and Nickerson, J. T. R., 1958, "Investigation of Bacterial Resistance of Packages" Report of QM Research Contract DA19-129-QM-758, MIT Cambridge, Mass.

<sup>2</sup> Szczablowski, J. W. and Rubinate, F. J., "Integrity of Food Packages," Modern Packaging, June 1965.

<sup>3</sup> Long, F. E., "Flexible Packages Now Withstand Heat Processing Temperatures of Foods", Packaging Engineering, March 1962.

TABLE 69

## STUDY OF FLEXIBLE PACKAGES UNDER VARIABLE TEMPERATURE CONDITIONS

## PERCENT PRODUCT WEIGHT CHANGE

## INITIAL EXPOSURE

STRUCTURE	Preconditioning Temperature			
	75°F.	0°F.	-40°F.	-70°F.
<u>50M-50F-.003"Marlex 6050</u>				
Avg. Range	+0.03 -0.12/+0.12	+0.07 -0.01/+0.16	-0.03 -0.09/+0.06	-0.12 -0.18/-0.04
<u>50M-50F-.002"Nylon 11</u>				
Avg. Range	+0.08 -0.02/+0.16	+0.04 -0.03/+0.13	-0.06 -0.17/+0.02	0.00 -0.05/+0.06
<u>50M-50F-.002"Vitel 409</u>				
Avg. Range	+0.04 -0.02/+0.11	+0.06 +0.02/+0.15	-0.03 -0.16/+0.03	-0.10 -0.13/+0.02

## ADDITIONAL EXPOSURE

STRUCTURE	Preconditioning Temperature			
	75°F.	0°F.	-40°F.	-70°F.
<u>50M-50F-.003"Marlex 6050</u>				
Avg. Range	+0.02 0/+0.03	-0.01 -0.01/-0.01	-0.04 -0.05/-0.02	+0.02 0/+0.03
<u>50M-50F-.002"Nylon 11</u>				
Avg. Range	+0.05 +0.04/+0.06	-0.01 +0.06/+0.08	-0.04 -0.04/-0.04	+0.02 +0.01/+0.04
<u>50M-50F-.002"Vitel 409</u>				
Avg. Range	0.00 -0.02/+0.02	+0.02 0.0/+0.03	0.00 -0.01/+0.02	-0.01 -0.20/+0.03

provide the concepts necessary for development of the integrity test method as follows:

a. The paperboard jacket, which contributes to the structural integrity of the food package, must not be separated from the pouch.

b. Under extreme circumstances in a military field operation, the food package could be totally immersed in contaminated water.

c. Aspiration of microorganisms into the flexible package could come from mechanical flexing of the package.

d. Microleakage, with entrance of gas-producing microorganisms, will result in significant swelling of the pouch, signaling a violated package.

e. The specific foods to be studied in this project are solids, i.e., ham, chicken and bacon. However in developing a "tentative" test method, a more useful food product consistency, chunks of food in a viscous sauce, was selected as a more difficult consistency to retain package integrity.

It must be emphasized that the major objective of this study was to establish a test method to evaluate specific flexible package structures after packing and processing and through the distribution and handling cycle to the ultimate user. The method was not devised as a quality control test of materials, of package fabrication methods, or of packing and sealing effects on the package.

Further, it should be stated that changes of material in the flexible laminate structure; changes in the protective jacket; changes in

sealing or joining; changes in size and shape, all contribute to produce a "new" or "different" flexible package. Hence, if a change is made it should be tested against a "standard" integrity testing method of sufficient replication to produce statistically significant data.

It is our opinion that the "tentative" integrity testing method which is reported in detail in this study can serve as a guideline to the development of a "standard" integrity testing method for flexible packages containing low acid, sterile foods intended for military feeding.

#### 15.3 Program of Work

The flow diagram of work in this study is shown in Figure 12.

#### 15.4 Development of the Integrity Test Method

Since a limited amount of data has been published in which flexible packages containing low acid, sterile foods have been tested for integrity in a reproducible test method, a schedule of handling abuse in military distribution was defined using the premise described above and agreed upon by the Project Officer, U. S. Army Natick Laboratories (See Figure 13).

##### 15.41 Development of the Bio-Test Procedure

###### Flexing Unit Development

Past experience in conducting bio-tests with rigid metal containers furnished the basic principles to follow in developing a bio-test procedure applicable to flexible pouches. The significant head space and high vacuum in the metal can represent a major difference in

Figure 12

STUDY OF MICROBIAL PENETRATION  
INTO FLEXIBLE PACKAGES

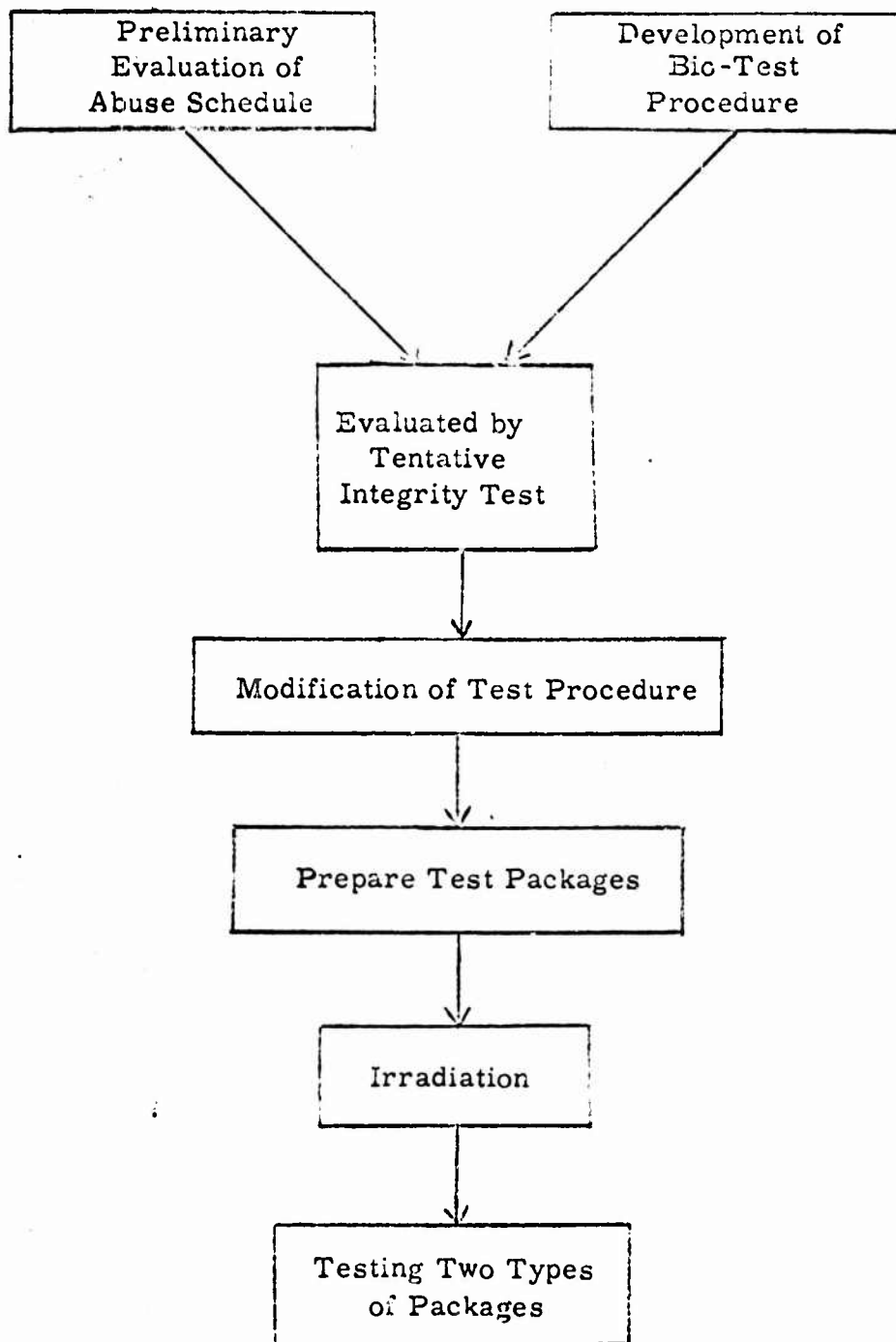
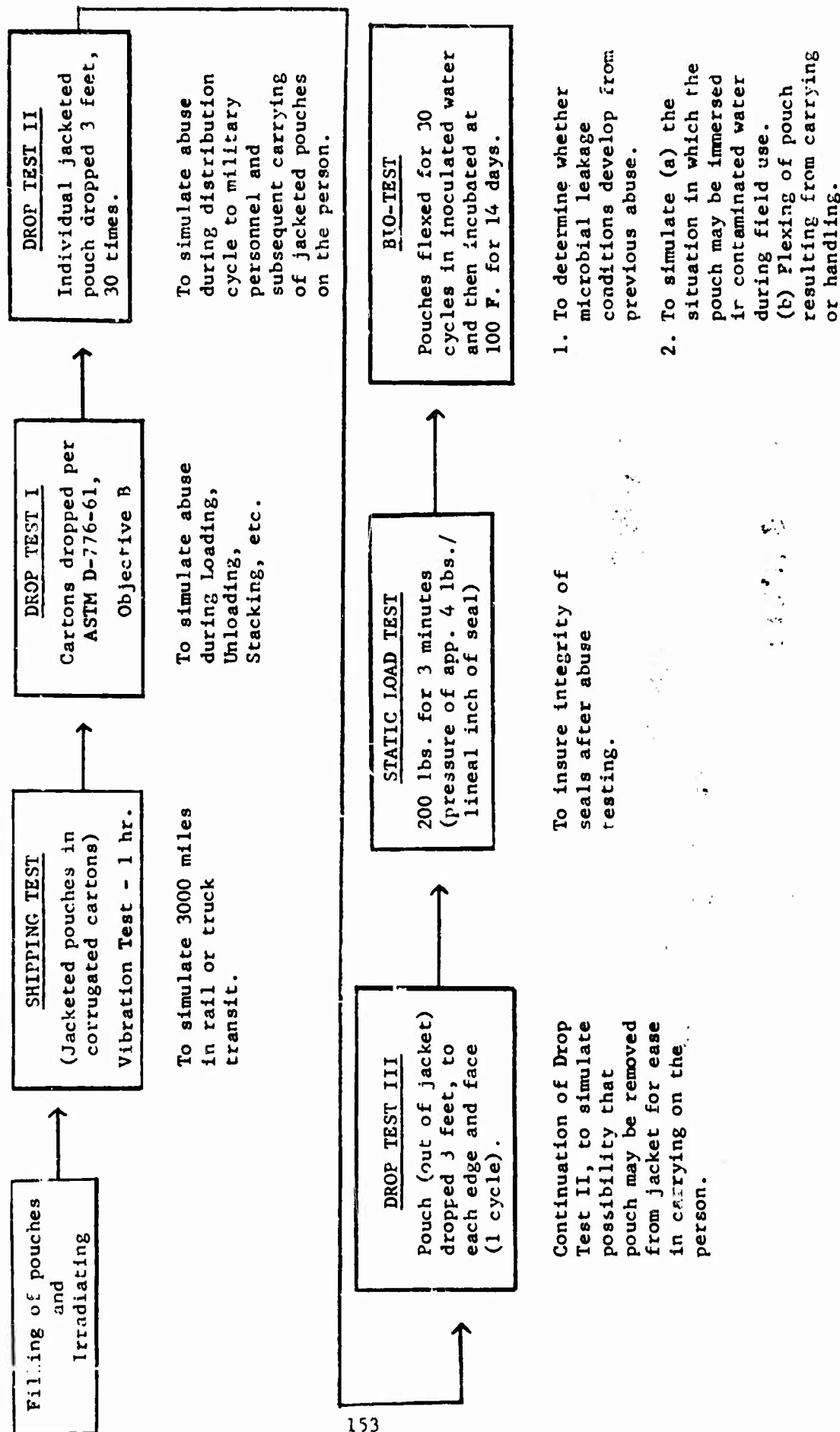


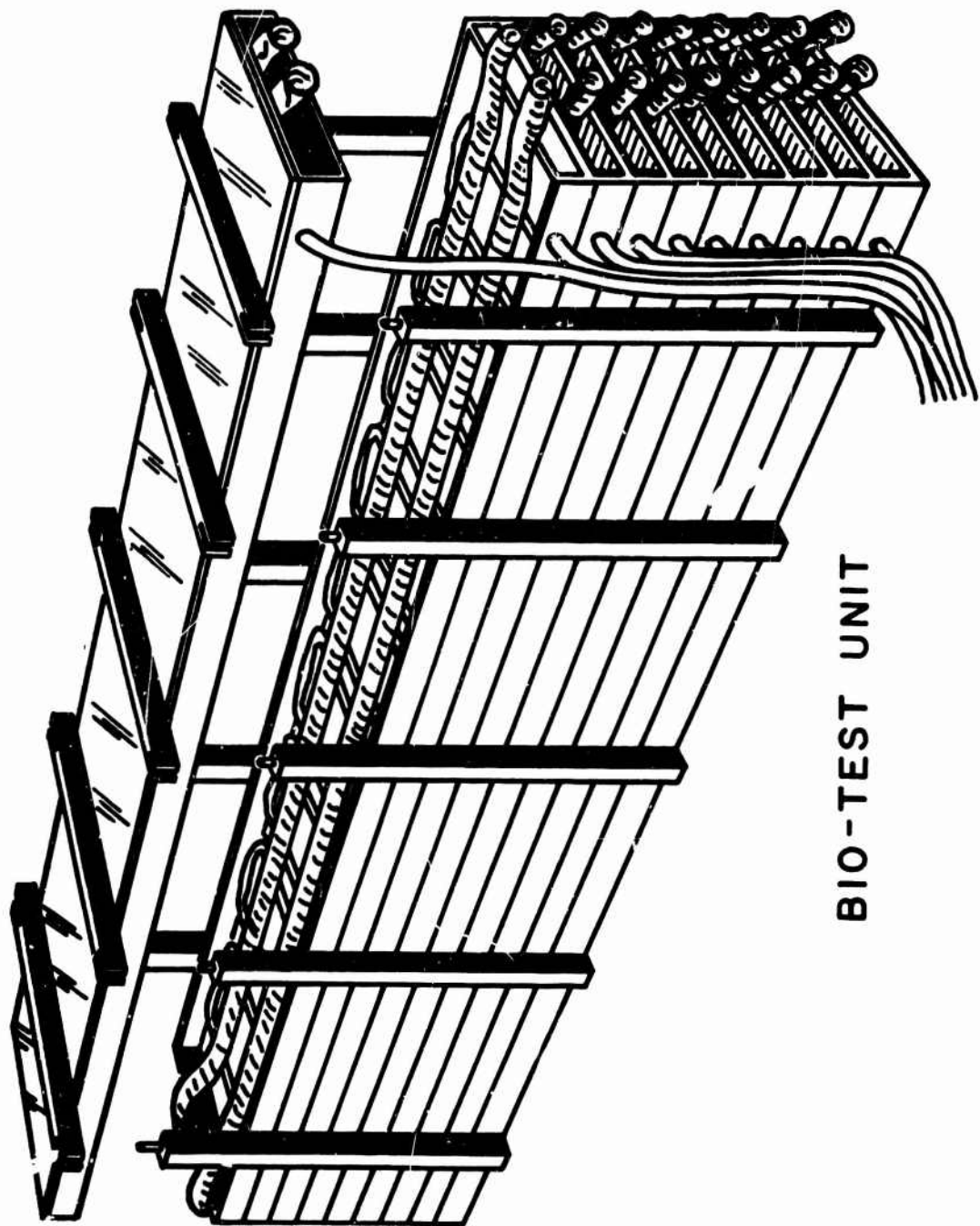
FIGURE 13

STUDY OF MICROBIAL PENETRATION OF FLEXIBLE PACKAGES

Schematic Diagram of Tentative Test Procedures



**FIGURE 14**



**BIO-TEST UNIT**

comparison to the flexible pouch. Whereas in the metal container, the vacuum is instrumental in drawing the bio-test organism through minute openings in a seam, it was considered that mechanically induced breathing action would be required to insure entrance to microorganisms through micro-openings in a flexible pouch.

Therefore, methods were examined for producing a flexing action to accomplish this purpose. Because pouches were sealed under vacuum, the possibility of inducing a flexing action by alternately pulling a higher external vacuum and releasing it was studied. An acrylic plastic chamber to hold a single pouch was constructed. This unit, containing a pouch of chicken-ala-king, was placed in a glass vacuum chamber for observation. Repeated cycles of vacuumizing to 26" and releasing failed to produce perceptible flexing action when the pouch was held either vertically or horizontally.

A device for applying pressure to portions of the pouch was next considered. It contained two pneumatic tubes, each 1.25" in diameter, which laid transversely across a row of pouches. The tubes were connected to a valving system permitting alternate pressurization under a controlled cycling rate. Although pressures attained in the pouches during flexing were not measured, more uniform application of pressure was attained when the tubes laid in metal channels rather than when the tubes contacted the pouches directly. Because of irregular transverse twisting action of the channels with 1.25" diameter tubes, 1.75" diameter tubes were eventually installed.

The experimental bio-test flexing unit consisted of a device which held twelve 4-1/2" x 7" pouches, side by side, in a horizontal position. Two pneumatic tubes, resting in metal channels, laid across the pouches. These were pressurized alternately (manifold pressure, 5 psig) at a rate of 0.15 min/cycle. The apparatus was immersed in a tank of water containing approximately  $10 \times 10^6$  cells of a 20-24 hour culture of Aerobacter aerogenes (CCC: CO-11). The inoculum was produced in a medium composed of 6 grams Bacto Nutrient Broth, 60 grams Sucrose and 360 grams Alaskan seed peas per 6 liters water contained in a 2 gal. F-style can and incubated at 35°C. Twelve liters of inoculum were usually strained to remove peas, then added to about 300 gallons of water. Inoculated water was continually agitated during bio-testing by means of compressed air entering at the bottom of the tank through a perforated pipe. The flexing treatment tended to aspirate bacteria, located at a point of microleakage, into the pouch.

Based upon experimental results obtained with the prototype bio-test unit, a full scale flexing unit consisting of nine tiers, each being comparable to the prototype, was constructed. (See Figure 14.) This unit was immersed in a commercially available galvanized tank, approximately 7' long by 3' wide, 2-1/2' deep, containing inoculated water prepared as previously described. For agitation of the inoculum, the tank was supplied with compressed air through a U-shaped perforated pipe situated on the bottom, running the full length of each side and across one end of the tank.

Calibration measurements to permit reproducibility in both construction and operation of the bio-test unit were performed. A cathetometer, capable of resolving 0.01 mm., was used to measure deflections at three points for each channel - at center and both ends. Strips of foam rubber (Sears, Roebuck Cat No. 24K8964P), 0.5" thick and extending from the outer edges of adjacent channels in each tier, were used in place of test pouches.

It was concluded that variations in channel deflections were due to machine variables and not to non-uniform compressive characteristics of the foam rubber (See Table 71).

Force-compression data for eight pieces of foam rubber tested on an Instron Testing Machine are given in Table 1. Neglecting the weight of the stainless steel channels (approximately 0.019 lbs. per square inch of contacting surface), a 10.0 to 10.5 mm. deflection would be expected when manifold pressures were changed from 0 to 4.5 psig. Calculated deflection agreed with maximum observed deflections.

Deflection data for the nine tiers are shown in Table 2. Inherent characteristics of the channels may contribute to variations in deflections. For example, twisting of a channel by 2.5 degrees during a half cycle will produce a measurement error of 2 mm. Installation of the 1.75" tubes minimized the twisting effect. Further, the length of the channels permitted some bending. The weight of the channel will be non-uniformly distributed along the foam rubber when its tube is

TABLE 70

Controlled Abuse Tester Channel Deflections, mm.

<u>Tier</u>	<u>Side I</u>			<u>Side II</u>		
	<u>Left*</u>	<u>Center</u>	<u>Right</u>	<u>Left</u>	<u>Center</u>	<u>Right*</u>
1 (top)	8.85	8.85	8.15	9.55	9.25	8.70
2	7.85	7.65	8.30	8.00	7.65	7.15
3	8.90	9.80	10.30	8.90	7.90	8.15
4	9.90	9.60	9.65	9.80	9.25	8.75
5	8.00	8.00	7.75	9.10	8.35	8.95
6	9.25	9.05	8.80	7.60	8.15	8.50
7	10.20	10.40	9.75	8.90	8.65	8.30
8	9.40	8.70	9.10	8.50	8.45	8.30
9	9.70	9.45	8.65	8.95	7.00	6.40
Average	9.12	8.17	8.94	8.81	8.29	8.13

\* Manifold end

Maximum manifold pressure 4.5 p.s.i. on both sides.

TABLE 71

Force-Compression Data for Sears, Roebuck  
Catalog No. 24K8964P Latex Padding, 1/2 Inch Thick

<u>Deflection, mm.</u>	<u>5.1</u>	<u>7.6</u>	<u>10.2</u>	<u>Deflection for 4.5 p.s.i.</u>
Sample 1	0.80 psi	1.5 psi	4.7 psi	10.0 mm.
2	.83	1.5	4.5	10.2
3	.83	1.5	4.8	10.0
4	.85	1.6	4.2	10.3
5	.83	1.4	3.9	10.5
6	.80	1.5	4.4	10.3
7	.85	1.6	4.2	10.3
8	.88	1.6	2	10.3

deflated. The rubber will be more compressed at points of load concentrations and will consequently undergo less compression as its tube is inflated.

The channel in the fifth tier on side II is known to be bent in a manner to produce variations in deflection (Table 70).

#### Control Pouch Piercing

Flexible pouches, of the type of laminate structure and containing food under test, must be suitably pierced, as controls, and included with each batch of test pouches in the bio-test procedure in order to establish assurance that contaminated water in the bath will indeed inoculate the food and produce swollen pouches. Our original premise was established on the basis that the hole should be ten to twenty times the size of an average bacteria dimension i.e., 0.5 microns. Experimentally, we found, in the food product we used as a "standard," chicken-ala-king, that much larger holes were necessary to cause an 85-99% inoculation of the control pouches.

The method of piercing adopted for all control pouches consisted of holding a No. 14 sewing needle in hemostatic forceps so that approximately 1/16" of the needle point was able to puncture and enter the pouch.

During the development of the bio-test procedure, approximately 386 (R-2) retort type flexible packages containing chicken-ala-king were utilized. (These pouches were furnished by the General Equipment and Packaging Laboratory, U. S. Army Natick Laboratories for use in developing of the "tentative" integrity test.)

Puncture location for the control pouches was approximately 1/2" from the top seal and mid-point between the side seals. This location was selected as a site of high stress and potential flexural fatigue in the pouch as well as for convenience in manipulating the pouch during puncturing.

A total of 386 punctured pouches of chicken-skin were used in the development of operating procedure for the bio-test apparatus, e.g., determining satisfactory flexing-tube pressures, cycles and running periods, inoculated bath population levels, effect on leakage of hole location and size, etc. Of this number, 89% became contaminated during flexing. Of the 11% which failed to leak, hole sizes ranged from 33 to 160 microns (for non-round holes, the longer dimension was taken into consideration); the median for the group was 98 microns and the mean, 102 microns. One group of 24 pouches having holes ranging from 330 to 1950 microns showed two non-leakers (this group was not included in the 386). Observations of the non-leakers suggested that the food product plugged the holes, preventing entrance of bacteria. Microscopic observations of the non-leakers in the group of 386 frequently suggested the same situation.

#### 15.42 Preliminary Evaluation of Handling Abuse

Concurrently with the development of the bio-test procedure, an initial experiment was run to assess the effects of the test method upon three types of flexible packages under study in this contract as follows:

a. Two shipping cartons containing 96 unirradiated pouches of the 50M-50F-.003" Marlex 6050, 50M-50F-.002" Nylon 11, 50M-50F-.002" Vitel 409 and R-2 (a standard pouch structure utilized by the U. S. Army Natick Laboratories in their work on flexible packages for retorted foods) structures were filled with water, sealed, assembled with the pouch glued in the center of the protective jacket, and positioned in shipping cartons (48 flexible packages/cartons).

b. Water was used to simulate the most difficult type of food to package since low viscosity products exert a greater inertial force against the pouch seal structures than high viscosity products.

c. All of the flexible packages were subjected in sequential steps to the test method (See Figure 13) with the bio-test omitted.

The failure (macro-leaks) rate at each step in the test procedure is shown below along with the over-all failure rate:

	<u>50M-50F-.002"</u> <u>Vitel 409</u>	<u>50M-50F-.003"</u> <u>Marlex 6050</u>	<u>50M-50F-.002"</u> <u>Nylon 11</u>	<u>R-2</u> <u>Retort</u>
	Failure Rate %			
Shipping Test	0	0	0	0
Drop Test I	25	27	1	0
Drop Test II	63	70	1	1
Drop Test III	49	50	2	0
Static Load	45	0	0	0
Over-all Failure Rate %	88	88	5	1

TABLE 72

PRELIMINARY EVALUATION OF TEST METHOD

Pouch Material	50M-50F-.002" Vitel 409	50M-50F-.003" Marlex 6050	50M-50F-.002" Nylon 11	R-2 Retort
Product	Water	Water	Water	Water
No. of pouches subjected sequentially to each step in the test	96	96	96	96
<u>Shipping Test</u>	<u>Number and Type of Failures</u>			
<b>DROP TEST I</b>				
Bottom Seal Junctionure	5	23		
Top Seal Junctionure	13	2		
Side Seal Junctionure	4		2	
Top Seal Crease		1		
Body Flexure	<u>2</u>			
Failures	24	<u>26</u>	<u>2</u>	<u>0</u>
<b>DROP TEST II</b>				
Bottom Seal Junctionure	14	33		
Top Seal Junctionure	15	2		
Side Seal Junctionure	8	13	1	1
Top Seal Crease		1		
Body Flexure	<u>2</u>			
Failures	39	<u>49</u>	<u>1</u>	<u>1</u>
<b>DROP TEST III</b>				
Bottom Seal Junctionure	6	6	1	
Top Seal Junctionure	3	1		
Side Seal Junctionure	8	2	1	
Body Flexure	<u>4</u>	<u>1</u>		
Failures	21	10	<u>2</u>	<u>0</u>
<b>STATIC LOAD</b>				
Side Seal Junctionure	1	0	0	0
<b>TOTAL</b>	<u>85</u>	<u>85</u>	<u>5</u>	<u>1</u>

The flexible structures that failed were inspected to determine the area of failure at each step in the test procedure. These data are presented in Table 72. As shown in Table 72, virtually all failures occurred at the junctures formed between the seal area and body of the pouch.

15.43 Preliminary Evaluation of the Test Method

The preliminary evaluation of the test method was continued as follows:

a. Commercially available 4 oz. packages of frozen chicken-ala-king were procured, the contents removed from the packages, and inserted in the 50M-50F-.003" Marlex 6050 and 50M-50F-.002" Nylon 11 pouches, which were then sealed on a Flex-Vac 69 machine. (The 50M-50F-.002" Vitel 409 was not included, based upon its performance in the pack test and the previous test).

These pouches were assembled in jackets, cartoned, and shipped (frozen) to the U. S. Army Natick Laboratories, for irradiation at a minimum dose of 4.5 megarads.

b. The irradiated, packaged, chicken-ala-king was then returned to the CCC Technical Center for testing. In the flexible packages under study, the chicken-ala-king serving (4 oz.) gave a package thickness dimension less than the solid products, ham, chicken, and bacon. Thus, fifty-four (54) flexible packages were needed to tightly pack the shipping cartons fabricated for this contract.

c. Two hundred sixteen (216) of each of the two types of flexible packages were subjected in sequential steps to the test method (See Figure 13), including the bio-test procedure. Fifty-four (54) R-2 packages (heat processed chicken-ala-king in retort pouches) were furnished by the General Equipment and Packaging Laboratory, U. S. Army Natick Laboratories for comparison purposes.

The failure rate at each step in the test procedure is shown below, along with the over-all failure rate:

	<u>50M-50F-.003"</u> <u>Marlex 6050</u>	<u>50M-50F-.002"</u> <u>Nylon 11</u>	<u>R-2</u> <u>Retort</u>
	Failure Rate %		
Shipping Test	0	0	0
Drop Test I	9	0	0
Drop Test II	36	0	15
Drop Test III	22	0	15
Static Load	6	0	0
Bio-Test (per cent swollen)	17	15	3
Over-all failure rate %	90	15	33

The flexible structures that failed were inspected to determine the area of failure at each step in the test procedure. These data are presented in Table 73. In comparing these results with the results obtained when water was the product packaged (See Section 15.42), the premise that the lower viscosity product will produce a greater rate of macro failures was substantiated. However, fatigue at the seal

juncture can permit entry of gas forming organisms, thus the over-all failure rate is high. Further, the R-2 retort pouches had a significant amount of additional handling prior to use in this test, hence, their failure rate was accentuated.

#### 15.44 Modification of the Packaging System and Test Method

The results of the preliminary evaluations of handling abuse and of the integrity test method showed that further large scale testing of the 50M-50F-.003" Marlex 6050 would be fruitless. In addition, the second highest failure rate in the handling abuse resulted from Drop Test III in which the flexible pouches were removed from the protective jacket.

These results were reviewed in a meeting of U. S. Army Natick Laboratories and CCC personnel. The following decisions were made at the meeting:

a. The flexible pouch should not be removed from the protective jacket during military distribution until actually ready for usage; hence, Drop Test III should be eliminated from the integrity test method procedures.

b. Provision should be made to discourage removal of the pouch from the jacket in field handling.

In accordance with those decisions, it was determined that the most direct method of providing for (b) above and enhancing the performance of the flexible packages, without developing a radically new concept, was to glue the seal area on all four sides to the protective jacket.

TABLE 73

PRELIMINARY EVALUATION OF INTEGRITY TEST METHOD

	50M-50F-.003" Marlex 6050	50M-50F-.002" Nylon 11	R-2 Retort
No. of Packages	216	216	54
<u>Shipping Test</u>	<u>Number and Type of Failures</u>		
<b>DROP TEST I</b>			
Bottom Seal Junctionure	14	0	0
Top Seal Junctionure	4	0	0
Side Seal Junctionure	1	0	0
Body Flexure	<u>1</u>	<u>0</u>	<u>0</u>
Failures	20	0	0
<b>DROP TEST II</b>			
Bottom Seal Junctionure	68	0	0
Top Seal Junctionure	1	0	8
Side Seal Junctionure	<u>2</u>	<u>0</u>	<u>0</u>
Failures	71	0	8
<b>DROP TEST III</b>			
Bottom Seal Junctionure	29	0	0
Top Seal Junctionure	3	0	6
Body Flexure	<u>0</u>	<u>0</u>	<u>1</u>
Failures	32	0	7
<b>STATIC LOAD</b>			
Bottom Seal Junctionure	5	0	0
Side Seal Junctionure	<u>1</u>	<u>0</u>	<u>0</u>
Failures	6	0	0
<b>TOTAL FAILURES</b>	129	0	15
<b>BIO-TEST</b>	66 samples plus 7 "controls" (pierced) (pre- punctured) of above remaining 87 pouches tested. Of 66 test samples, 11 swelled. Of 7 "control" samples, 5 swelled.	198 samples plus 18 "con- trols" (pierced) (pre- punctured) tested. Of 198 test samples, 29 swelled. Of 18 "control" samples, 18 swelled.	33 samples plus 2 "controls" of above remaining 39 pouches tested. Of 33 test samples 1 swelled. Of 2 "controls" 2 swelled.

Forty-eight (48) irradiated flexible pouches of the 50M-50F-.003" Marlex 6050 and ninety-six (96) unirradiated 50M-50F-.002" Vitel 409 structures were filled with 5 oz. of water and sealed. The pouches were placed in protective jackets which had ~~hot melt~~ adhesive applied to both surfaces of the jacket that were in contact with the pouch seal area. The adhesive application was as wide as the seal area and extended around the entire periphery of the pouch. After the pouches were enclosed in the jackets, the assembly was heat sealed along all four edges with a Sentinel heat sealer.

The flexible pouches were subjected to the first 5 test procedures, including Drop Test III, so that the results would be comparable to the testing discussed in Section 15.42.

The significant reduction in failure rate that is attributable to supporting the seal junctures by edge gluing the pouch to the jacket is readily apparent in comparing the performance summary below with the data shown in Section 15.42 (See Table 74):

	<u>50M-50F-.003"</u> <u>Marlex 6050</u>	<u>50M-50F-.002"</u> <u>Vitel 409</u>
	Failure Rate %	
Shipping Test	0	0
Drop Test I	4	1
Drop Test II	2	10
Drop Test III	18	23
Static Load	0	2
Over-all failure rate %	23	30

TABLE 74

TESTING OF MODIFIED PACKAGES

	50M-50F-.003" Marlex 6050 <u>(pouch edge glued to jacket)</u>	50M-50F-.002" Vitel 409 <u>(pouch edge glued to jacket)</u>
<u>Shipping Test</u>	<u>Number and Type of Failures</u>	
DROP TEST I		
Bottom Seal Juncture	1	1
Side Seal Juncture	0	0
Side Seal Creepout	0	0
Top Seal Juncture	1	0
Top Seal Crease	0	0
Body Flexure	<u>0</u>	<u>0</u>
Failures	2	1
DROP TEST II		
Bottom Seal Juncture	0	2
Side Seal Juncture	1	3
Top Seal Juncture	0	2
Body Flexure	<u>0</u>	<u>2</u>
Failures	1	9
DROP TEST III		
Bottom Seal Juncture	6	3
Side Seal Juncture	1	3
Top Seal Juncture	0	3
Body Flexure	<u>1</u>	<u>9</u>
Failures	8	18
STATIC LOAD		
Side Seal Juncture	0	1
TOTAL FAILURES	11	29

## 15.5 "Tentative" Package Integrity Test Method

### 15.51 Testing of Two Types of Packages

Based upon the results obtained in the development of the test method, a "tentative" package integrity test method was adopted for the major testing program, as shown in Figure 15.

Two types of packages were evaluated, i.e., the 50M-50F-.003" Marlex 6050 pouch, edge sealed in the protective jacket and the 50M-50F-.002" Nylon 11 center glued in the protective jacket. The testing of these was conducted as follows:

a. Approximately 3,000 each of the two types of flexible packages were packed in accordance with the procedure shown in Section 15.43 (a and b) and tested in the procedure shown in Figure 15.

b. Four hundred eighty (480) R-2 flexible pouches containing retorted chicken-ala-king were furnished by the General Equipment and Packaging Laboratory, U. S. Army Natick Laboratories to be tested in the procedure parallel with the two packages under study in this work.

c. The bio-test procedure, carried out as described in 15.41, permitted testing one hundred eight (108) pouches per load. Of these, nine (9) pouches were pierced as controls, using one (1) such pouch per tier in random location. As indicated in Table 77, the tank was drained and fresh water plus new inoculum added after each series of approximately five loads.

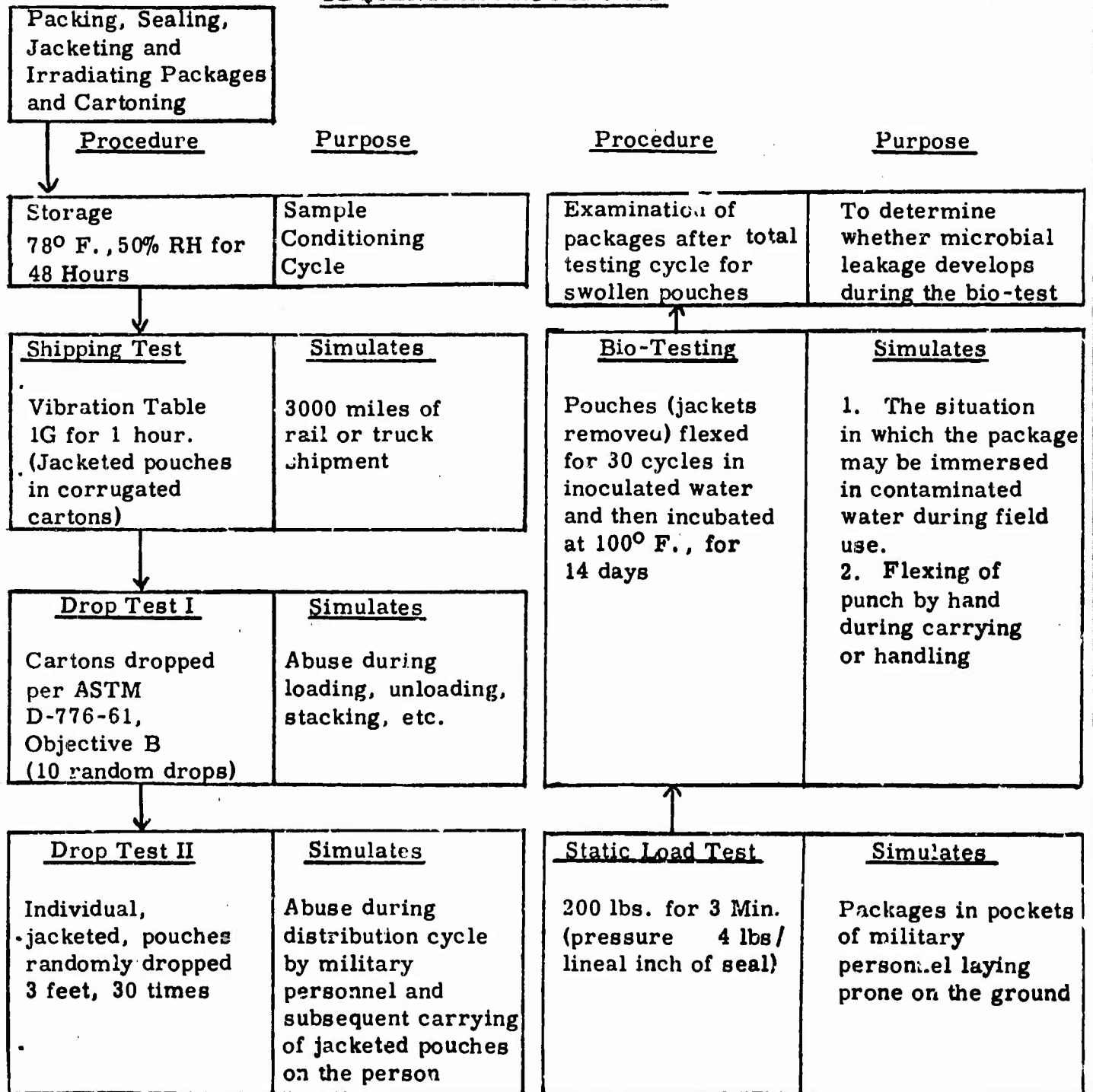
The principal results of the integrity testing of the two types of packages are shown below:

Figure 15

STUDY OF MICROBIAL PENETRATION  
INTO FLEXIBLE PACKAGES

"TENTATIVE" INTEGRITY TEST METHOD

SEQUENTIAL PROCEDURES



	50M-50F-.003" <u>Marlex 6050</u>	50M-50F-.002" <u>Nylon 11</u>	R-2 <u>Retort</u>
	Failure Rate %		
Shipping Test	0	0	0
Drop Test I	0	0	0
Drop Test II	17	1	4
Static Load	1	1	1
<u>Bio-Test (per cent swollen)</u>	<u>40</u>	<u>17</u>	<u>5</u>
Over-all failure rate %	51	17	10

The detailed results of the test are shown in Tables 75-79.

Approximately 1,000 each of the two types of flexible packages containing irradiated chicken-ala-king were not included in the test. The packages were shipped to the U. S. Army Natick Laboratories for their use in testing these packages in the "combat course" testing program.

#### 15.6 Discussion of Test Method Results

The premise for the development of this "tentative" flexible package integrity test method was to establish a "reproducible" test method comparable to typical military food distribution and usage. Previous performance testing of the R-2 retorted flexible package had been accomplished by the U. S. Army General Equipment Test Activity, Fort Lee, Virginia for the General Equipment and Packaging Laboratory, U. S. Army Natick Laboratories. The flexible pouch structure in those tests

TABLE 75

"TENTATIVE" INTEGRITY TEST RESULTS

	50M-50F-.003" Marlex 6050 (Pouches Edge Glued)	50M-50F-002" Nylon 11 (Pouches Center Glued)	R-2 Retort (Pouches Center Glued)
Number of Packages of Chicken-ala-king	2052	2052	480
Shipping Test	<u>Number and Type of Failures</u> (Not Inspected After Test)		
DROP TEST I	0	0	0
DROP TEST II			
Bottom Seal Junctionure	310	0	0
Side Seal Junctionure	20	0	0
Top Seal Junctionure	19	1	0
Body Flexure	<u>0</u>	<u>0</u>	<u>18</u>
	349	1	18
STATIC LOAD			
Bottom Seal Junctionure	4	0	0
Side Seal Junctionure	0	0	0
Top Seal Junctionure	0	2	5
Body Flexure	<u>0</u>	<u>0</u>	<u>0</u>
	4	2	5
BIO-TEST			
Number of Positive Swells	645*	329**	20***
FAILURE RATE	40%	17%	5%

\* 90 pierced controls were used in addition to test pouches; 90% swelled

\*\* 90 pierced controls were used in addition to test pouches; 97% swelled

\*\*\* 38 pierced controls were used in addition to test pouches; 95% swelled

TABLE 76

BIO-TEST, FLEXIBLE POUCHESVariable 50M-50F-.003" Marlex 6050

<u>Load No.</u>	<u>Total Pouches</u>	<u>Growth in Pierced Controls</u>		<u>Growth in Abused Pouches</u>		<u>Bact. Population (Bath) Per ML</u>
		<u>Pos.</u>	<u>Neg.</u>	<u>Pos.</u>	<u>Neg.</u>	
1	108	9	0	40	59	*18x10 <sup>6</sup>
2	108	9	0	45	54	
3	108	0	0	48	60	
4	108	9	0	35	64	
5	108	8	1	42	57	10x10 <sup>6</sup>
6	108	3	1	37	62	*12x10 <sup>6</sup>
7	108	7	2	35	64	
8	108	0	0	46	62	
9	108	0	0	45	63	
10	108	0	0	38	70	
11	108	7	2	37	62	
12	108	6	3	39	60	12x10 <sup>6</sup>
13	108	9	0	29	70	*26x10 <sup>6</sup>
14	108	9	0	51	48	
15	108	0	0	45	63	
16	<u>76</u>	<u>0</u>	<u>0</u>	<u>33</u>	<u>43</u>	
TOTALS	1696	81(90%)	9	645(40%)	961	

\* Tank emptied and fresh water and inoculum added

TABLE 77

50M-50F-.002" Nylon 11

<u>Load No.</u>	<u>Total Pouches</u>	<u>Growth in Pierced Controls</u>		<u>Growth in Abused Pouches</u>		<u>Bact. Population (Bath) Per ML</u>
		<u>Pos.</u>	<u>Neg.</u>	<u>Pos.</u>	<u>Neg.</u>	
16	32	0	0	6	26	30x10 <sup>6</sup>
17	108	0	0	16	92	
18	108	0	0	16	92	
19	108	0	0	18	90	
20	108	9	0	14	8	
21	108	9	0	9	90	29x10 <sup>6</sup>
22	108	8	1	16	83	*31x10 <sup>6</sup>
23	108	9	0	18	81	
24	108	0	0	18	90	
25	108	0	0	19	89	
26	108	9	0	24	75	
27	108	8	1	16	83	28x10 <sup>6</sup>
28	108	9	0	18	81	*22x10 <sup>6</sup>
29	108	9	0	17	82	
30	108	0	0	20	88	
31	108	0	0	19	89	
32	108	0	0	14	94	
33	108	8	1	19	80	
34	108	9	0	18	81	
35	<u>71</u>	<u>0</u>	<u>0</u>	<u>14</u>	<u>57</u>	26x10 <sup>6</sup>
TOTALS	2047	87(97%)	3	329(17%)	1628	

\* Tank emptied and fresh water and inoculum added

TABLE 78

Retort Pouches: Chicken-ala-King

<u>Run No.</u>	<u>Total Pouches</u>	<u>Pierced Controls</u>		<u>Abused Pouches</u>		<u>Bact. Population (Bath) (per ML)</u>
		<u>Pos.</u>	<u>Neg.</u>	<u>Pos.</u>	<u>Neg.</u>	
1	108	8	1	7	92	15x10 <sup>6</sup>
2	108	8	1	2	97	
3	108	9	0	7	92	
4	108	9	0	4	95	13x10 <sup>6</sup>
5	<u>24</u>	<u>2</u>	<u>0</u>	<u>0</u>	<u>22</u>	
<b>TOTALS</b>	456	36	2	20	398	

consisted of 0.5 mil polyester - 0.35 mil aluminum foil - .003 mil polyolefin. A leakage rate of less than 10% was found in that severe testing, which was equivalent to 10 days field use. However, the package performance for actual military utilization has been established as one day of field use.<sup>1</sup>

Based upon the work in developing and applying the "tentative" flexible package integrity test, we have reached the following conclusions:

- a. The failure rate of the R-2 retort type package in this test is approximately equal to the rate found in the testing performed under the simulated combat conditions noted above.
- b. Since the military requirement established to date had been for one day of field use, it appears likely that the "tentative" integrity test procedure developed in this project is too severe.
- c. Additional work to correlate simulated combat testing with the "tentative" integrity test method is necessary to establish an "acceptable" test method.
- d. Since the pouch structure consisting of 50M-50F-.002" Nylon 11 did not perform as well as the R-2 retort package, increasing the Nylon 11 film thickness to .003 should give a durability performance equivalent to the R-2 retort pouch. An alternative approach would consist of edge gluing or supporting the seal-body juncture of the pouch in the protective jacket.

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<sup>1</sup> J. W. Szczablowski and F. J. Rubinate, "Integrity of Food Packages," Modern Packaging, June 1965, p. 131-134.

e. The very high failure rate (40%) of 50M-50F-.003" Marlex 6050 packages indicates that increases in the film thickness and seal support will not provide a satisfactory package for low viscosity foods. A more promising approach to the use of a linear polyethylene food contacting film would be a modification of the resin through copolymerization or blending.

f. Support of the pouch seal areas to reduce the stress on seal-body juncture should be incorporated in all future package design developments since practically all failures occur at the seal area juncture.

g. The bio-test procedure must be included in package integrity testing as the final test to confirm or deny the presence of micro-openings in the type of packages which will permit the entrance of microorganisms into the flexible pouch. Since excessive abuse can be imposed upon flexible packages without visual evidence of damage that can be observed on abused containers of other types.

h. Ideally, a specific type of flexible package for a specific food product should be tested for integrity in the "tentative" test method. For example, a solid piece of meat with a small amount of liquid, vacuum packed in a flexible package, would offer less hydraulic stress on the seal area juncture during handling abuse.

TABLE 79

IRRADIATED POUCH PACK TEST

SUMMARY OF POUCH LEAKAGE LOCATION DURING TEST. (SWELLS ONLY)  
(Pierced control pouches not included)

50M-50F-.003"  
MARLEX 6050  
(11 pouches)

50M-50F-.002"  
NYLON 11  
(29 pouches)

No. of leaks detctd. in this area	No. of pchs inv. in these leaks	% of total leaks det. in this area	Break- down of leaks	% break- down of leaks	AREA OF POUCH WHERE LEAKAGE WAS DETECTED	Break- down of leaks	% of total leaks det. in this area	No. of pchs inv in these leaks	No. of leaks det. in this area
28	2.4	80%			TOP SEAL-AREA		2.4%	5	5
	16	56%			Seal jun fracture	60%		3	
	12	44%			Seal channel crease	40%		2	
179	5	15%			SIDE SEAL AREA		33%	6	7
	4	80%			Seal jun fracture	86%		6	
	1				Seal channel crease	1.4%		1	
2	2	5%			BOTTOM SEAL AREA		38%	7	8
	1	50%			Seal jun fracture	25%		2	
	1	50%			Seal channel crease	75%		6	
					POUCH BODY AREA		5%	1	1
					Flexual pin hole	100%		1	
35	35				COMBINED POUCH TOTAL			21	21
	21	60%			Seal jun fracture	52%		11	
	14	40%			Seal channel crease	43%		9	
					Flexual pinhole	5%		1	

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## APPENDIX A

### Flexible Packaging Division

Number STM-8  
Issued 8-28-43

NAME: BOND STRENGTH DETERMINATION

PURPOSE: The purpose of this test is to determine the bond strength or resistance to separation of one flexible ply from another when bonded together by an adhesive and where the adhesive is the inner member ply.

EQUIPMENT:

- (1) Scott Tester, Model X-5 (Henry L. Scott Co., Providence, R. I.).
- (2) Suter Tester (Alfred Suter, Textile Engineer, 200 Fifth Avenue, N. Y.).
- (3) Any other mechanically operated device as a suitable means of holding this specimen at a uniform rate of pull and means of recording the resulting strength values.

DESCRIPTION: These are mechanically actuated machines in which the specimen is stressed by a uniform movement of the pulling clamp to which one end of the specimen is attached, the other end being held in a clamp attached to a weighing device based on the principle of the pendulum. The rate of pull is 12 inches per minute. The pendulum device automatically registers the pull in grams required to separate the plies on a calibrated scale.

(1) Capacity: The equipment has various means for changing the total loading, or larger size testers may be used, providing the loading is kept to at least 25 per cent of the maximum measurable load. One-inch specimens should be used whenever possible but it is permissible to use 1/2 inch wide specimens to come within the loaded range of the maximum. Under no circumstances shall specimens be tested with width less than 1/2 inch. In every case, the results shall be calibrated and reported in terms of one inch of width of bond.

(2) Calibration and Adjustment: Before each bond test is made, the pendulum device must be set at zero to give proper readings. The testers must be calibrated once a month in the following manner:

Level the machine accurately in both directions and clean the mechanisms to insure that it moves freely. Apply various load loads to the clamp actuating the indicating mechanisms and note the scale readings when the load and mechanism come gently into an equilibrium position. This can be conveniently done with the pendulum - type of tester by wedging up the pawls holding the pendulum with a small piece of paper bent double, suspending the test weights to the upper clamp and allowing the pendulum to come to equilibrium from the direction in which it moves when the load is applied to it. The lower jaw should not be used for supporting the test weights during calibration. Make a record of deviations from the indicated readings and apply corresponding corrections to the test results.

**TEST SAMPLES:**

(a) The test sample shall be representative of the material being tested.

(b) Five samples cut an inch wide taken from the longitudinal or machine direction of the material being tested shall be tested. The samples shall be taken across and width of the web so that each side and the middle areas will be tested.

(c) The test sample shall be 5 inches in length.

(d) The utmost care shall be exercised in cutting strip samples to prevent nicks and tears in the edges which might give rise to premature tears. The edges shall be parallel to within 2% of the width.

**CONDITIONING:**

All test samples shall be tested at standard room temperatures. Standard room temperature is defined as an atmosphere of unspecified relative humidity at a temperature in the range of 20 to 30°C (68-86°F).

**PROCEDURE:**

(a) The plies of the test sample are separated slightly by the action of an active solvent or by the application of heat.

(b) The free ends are then placed and anchored in the two clamps, taking care that the sample is lined up parallel to the clamps.

(c) The "tail" of the sample must be placed down and towards the testing machine. Both plies must be separated from the "tail" at a 90° angle.

(d) After the loose ends are secured in the jaws, release the holding lever to start the test.

(e) Observe the readings on the scale. If the reading goes beyond the limits of the scale, an additional weight, provided, must be attached to the pendulum.

**CALCULATIONS:**

(a) Record the bond strength as grams per linear inch.

(b) Results of the 5 strips tested shall not vary more than 5% from the average.

(c) If bond strengths of the 5 samples vary more than 5% from the average another 5 samples shall be taken for test.

**REMARKS:**

(a) With any paper structure look for a tear of the sheet fibres - this indicates satisfactory bond.

(b) Some light gauge materials are supported by Scotch tape in the clamping jaws to prevent tearing or slipping.

**REFERENCES:**

TAPPI, Testing Methods. Recommended Practices, Specifications, T-404-M-41.

## APPENDIX B

### Flexible Packaging Division

Number STM-13  
Issued

NAME: SEAL STRENGTHS

PURPOSE: To determine the seal strength of heat sealable materials and coatings, etc.

EQUIPMENT: (1) Wrap-ade sealer with flat metal chrome plated jaws (both heated) Model No. 80, set for 1/2 second dwell time, or Packaging Industries, Ltd. Sentinel sealer with variable dwell temperature and pressure.

(2) Standard Alfred Suter Tensile Tester, or equivalent, cross-head speed of 12 inches per minute.

(3) Laboratory hand laminator or draw down equipment.

PROCEDURE: (1) For Heat Sealable coatings, etc., prepare a draw down on the appropriate web material or coat by use of the hand laminator. Dry coating free of solvents; for heat sealable films or materials already heat seal coated proceed as in 2.

(2) Cut test sample into 5" width, approximately 2-1/2" long.

(3) Seal test samples together in the face to face structure with the heat sealing agent as the interface or sealing surface. Use type of sealer, dwell time, temperature or pressure as specified. See Remarks 3.

(4) Cool the sealed sample (between two cool metal plates) to room temperature and cut exactly into 1" widths.

(5) Insert the test sample into the jaws of the tensile tester so that one tongue of the test sample is in the upper jaw and the other is in the lower jaw with the sealed portion away from the operator and placed downward. Be sure the jaws of the tester are tight.

(6) Operate the appropriate controls on the tensile tester to determine the seal strength of each strip.

**RESULTS:**

(1) Report results in grams/inch of width (usually the average of five individual strips). Include type of sealer, temperature, dwell time and pressure where applicable.

(2) Indicate the optimum seal temperature for the test sample.

**REMARKS:**

(1) For materials which are heat sealable on both sides, use 50 gauge Mylar "C" as slip sheet to sandwich between jaws of sealer.

(2) Use of Sentinel sealer implies only top jaw heated. The top jaw is teflon coated and the bottom backing consists of 1/4" thick silicon rubber covered with a sheet of teflon coated glass cloth.

(3) To determine seal range, standard procedure is to use Sentinel sealer, 40 psi., 1/2 second dwell, and temperature in increments of 25°F from the lowest seal temperature to maximum temperature at which heat resistance of test materials is satisfactory.

APPENDIX C

Flexible Packaging Division

Number STM-29  
Issued 7-9-48

NAME: BURSTING STRENGTH OF PAPER

PURPOSE: To determine the bursting strength of paper.

EQUIPMENT: Model "C" electric Mullen Tester (B. F. Perkins & Sons, Inc.).

PROCEDURE:

- (1) Clamp full size sheet in the machine and force clutch handle to left. When paper bursts, throw handle to stop position.
- (2) Read results on gauge as pounds/sq. in.
- (3) Take ten readings on ten separate sheets.
- (4) Do not allow rubber diaphragm to inflate beyond top of clamp ring.

RESULTS: Report average results, giving maximum and minimum results.

## APPENDIX D

### Flexible Packaging Division

Number SIR-30  
Issued 11-28-58

NAME: TEAR RESISTANCE

PURPOSE: To measure the internal tearing resistance of papers.

EQUIPMENT: (1) Elmendorf Tear Tester.  
(2) Conditioning cabinet maintained at 50% R.H. and 73°F with circulating air to insure uniform conditions.

PROCEDURE: (1) Suspend test specimens in the humidity cabinet so that the conditioning atmosphere will have free access to all surfaces.  
(2) Check weight of samples at appropriate intervals usually 2-6 hours until there is no significant gain (1 part in 1000) in weight.  
(3) Cut 5 samples in both machine and transverse direction 3" to 4" and exactly 2-1/2" long. The number of sheets to a sample may be varied from 1 to 16 sheets depending on the strength of the material. For accuracy, the number of sheets to a sample should be adjusted to give readings between 20 and 60 on the scale.  
(4) Adjust equipment to zero and clamp test sheet (sheets) between the jaws.  
(5) Make initial slit with knife which is mounted on tester.  
(6) Release the pendulum and take reading.

RESULTS: (1) Report results in grams by the following equation:

$$\text{Grams/l sheet} = \frac{\text{average reading} \times 16}{\text{No. of sheets used in test.}}$$

(2) Report results of machine and transverse directions.

**REMARKS:**

(1) Duplicate determinations on different sets of samples from the same shipment and on different testing instruments should agree with each other within 7%.

(2) For thin gauge plastic sheeting, laminated films, foils, and papers, condition according to specification or determine at prevailing room conditions.

## APPENDIX E

### Flexible Packaging Division

Number STM-57  
Issued 6-3-49

#### NAME:

TENSILE PROPERTIES OF THIN PLASTIC SHEETS AND FILMS

#### SCOPE:

This method covers a test for use in determining the tensile properties of organic plastic in the form of relatively thin sheets or films (less than 0.020 in. in thickness), when tested under defined conditions of pretreatment, temperature, humidity, and rate of stressing by a constant rate of load method. It is not intended for use in testing rubber-like materials whose percentage elongation is greater than approximately 350 (Note).

(Note - Such materials may be tested in accordance with the Standard Methods of Tension Testing of Vulcanized Rubber (ASTM Designation D 412.))

#### EQUIPMENT:

The apparatus shall consist of the following:

(a) Testing Machine: Any suitable film testing machine of the constant rate of loading type. It should be equipped with a device for recording the tensile load carried by the specimen and recording the rate of jaw separation during the test. Both the stressing and recording mechanism should be essentially free from inertia lag at the specified rate of stressing, and frictional drag should be reduced to a minimum. The machine should indicate the load with an accuracy of plus or minus 2 per cent, or better. A device for varying the loading rate between approximately 50 and 300 lb. per min. should be included (Note 1).

Note 1: A machine of the tilting-table type has been found satisfactory.

(b) Grips: Flat, smooth steel grips should be used where possible and tests should be started with the grips separated a distance of 2 in. (Note 2).

Note 2: Other types of gripping surfaces may be required for some types of plastics. For example, file-faced grips or grips lined with abrasive paper have been found necessary for some materials. Also, it may be necessary in some cases to use an initial grip spacing of less than 2 in. Neither of these variations should be used unless it is impossible to obtain satisfactory results with the conditions recommended.

(c) Micrometers: Suitable micrometers, or other thickness gauges, reading to 0.0001 in. or less, for measuring the width and thickness of the test specimens.

(d) Paper Cutter: A suitable paper cutter or trimmer for trimming the specimens to the proper width. It is important that the cutting edges of the cutter be kept sharp and free from scratches or nicks.

(e) Conditioning Apparatus: Apparatus for maintaining suitable atmospheric conditions, as defined in the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618). Unless otherwise specified, the standard laboratory atmospheric conditions of 25± or - 1 C. (77± or - 2 F.) and 50± or - 2 per cent relative humidity is required.

#### **TEST SPECIMEN:**

(a) The test specimen shall consist of a strip of uniform width and thickness, and 5 in. or more in length.

(b) The width of the specimen shall be not less than 0.25 in. but not greater than 1.0 in. and shall be chosen so as to allow failure to occur well within the loading rate

which gives the proper rate of stressing shall be selected.

(c) A width-thickness ratio of greater than eight shall be used, if possible, since narrow strips emphasize the effect of edge strains or flaws incurred during cutting.

(d) The utmost care shall be exercised in cutting strip specimens to prevent nicks and tears in the edges which might give rise to premature fractures. The sheet shall be held firmly, and close to the knife edge. The blade shall be drawn through the sheet slowly but smoothly. The edges shall be parallel to within 2 per cent of the width.

(e) Gage marks shall be placed on the specimen in order to obtain reliable elongation data. (Note) This shall be done with a soft, fine wax crayon, or with India ink. Gage marks shall not be scratched onto the surface with a sharp instrument, because this causes weakness and premature fracture as a result of stress concentration.

Note: The use of gage marks is necessary because materials with appreciable elongation pull away from the grips. As the specimen elongates, the accompanying reduction in area results in a loosening of material just inside the grips. This moves back into the grips as further elongation and reduction in area take place.

(f) In testing materials which may be suspected of anisotropy, duplicate sets of test specimens shall be prepared having their long axis respectively parallel with and normal to the suspected direction of anisotropy.

#### CONDITIONING:

(a) Standard Laboratory Atmosphere. An atmosphere having a relative humidity of  $50 \pm 2$  per cent at a temperature of  $25 \pm 1^\circ\text{C}$ . ( $77 \pm 1.8^\circ\text{F}$ .) shall be the standard Laboratory Atmosphere.

(b) Standard Room Temperature. An atmosphere of unspecified relative humidity at a temperature in the range of 20 to 30 C. (68 to 86 F.).

(c) Standard Test Temperatures. When the effect of temperature on a property is to be determined or reported, the test temperatures shall be chosen from the following:

<u>Test Temperatures</u>	<u>Tolerance, plus or minus</u>
-70 C. (-94 F.)	2.0 C. (3.6 F.)
-55 C. (-67 F.)	2.0 C. (3.6 F.)
-40 C. (-40 F.)	2.0 C. (3.6 F.)
-25 C. (-13 F.)	2.0 C. (3.6 F.)
0 C. (32 F.)	1.0 C. (1.8 F.)
25 C. (77 F.)	1.0 C. (1.8 F.)
50 C. (122 F.)	1.0 C. (1.8 F.)
70 C. (158 F.)	1.0 C. (1.8 F.)
90 C. (194 F.)	1.0 C. (1.8 F.)
105 C. (221 F.)	1.0 C. (1.8 F.)

(d) Conditioning Prior to Test

(1) Standard Procedure: Test specimens 1/4 in. or less in thickness shall be conditioned in the Standard Laboratory Atmosphere (with adequate air circulation) for a minimum period of 40 hr. immediately prior to testing. Test specimens greater than 1/4 in. in thickness shall be conditioned in the Standard Laboratory Atmosphere (with adequate air circulation) for a minimum period of 88 hr. immediately prior to testing. The standard procedure shall be used, unless otherwise specified.

Note 1: Standard Procedure is generally satisfactory and recommended for use unless functional procedures are specified.

(2) Functional Procedure: A. - Test specimens shall be conditioned for a period of 48 hr. in a circulation air oven at a temperature of 50 + or - 3 C. (122 + or - 5.4 F.). The specimens shall be removed from the oven

and cooled to the Standard Room Temperature in a desiccator over anhydrous calcium chloride (or other suitable desiccant) for a period of at least 16 hr. immediately prior to testing.

Note 2: Functional Procedure A is recommended for use wherever the specific effects of drying are to be determined.

(3) Functional Procedure: B. - Test specimens shall be conditioned for a period of 96 + or - 2 hr. at a temperature of 35 + or - 2 C. (95 + or - 3.6 F.) in an atmosphere of 90% relative humidity the tolerances on humidity; being + or - 2 per cent for general purposes and + or - 1% for reference purposes.

Note 3: Functional Procedure B is recommended for use wherever the specific effects of exposure to severe atmospheric moisture are to be determined.

(e) Tests at Normal Temperatures

(1) Unless otherwise specified, materials conditioned according to the Standard Procedure shall be tested in the Standard Laboratory Atmosphere.

(2) Unless otherwise specified, materials conditioned according to Functional Procedure A shall be tested at Standard Room Temperature conditions. Not more than 1/2 hr. shall elapse between removal of the specimens from the desiccator and the start of the tests.

(3) Unless otherwise specified materials conditioned according to Functional Procedure B shall be tested in the same atmosphere (Section (d) - (3)).

(f) Tests at other Standard Test Temperatures (See Section (c)).

All materials shall be transferred to the test conditions within 1/2 hr. preferably

immediately, after completion of the conditioning. The specimens shall be conditioned at the test temperature for not more than 5 hr. prior to tests, and in no case less than the time required to insure thermal equilibrium.

(g) Selection of Conditioning Procedure

(1) In the case of materials covered by A.S.T.M. specifications, preference should be made thereto to determine the conditioning procedures to be used.

(2) In the case of all other materials, choice between the conditioning procedures should be preferably based on the one which gives the most reproducible test results.

(h) Report

(1) The report shall state the conditioning procedure used and the atmospheric conditions under which the tests were made.

**NUMBER OF TEST SPECIMENS:**

(a) At least ten specimens shall be tested for each sample in the case of isotropic materials.

(b) Twenty specimens, ten normal to and ten parallel with the principal axis of anisotropy, shall be tested for each sample in case of anisotropic materials.

(c) Results on specimens which break at some obvious chance flaw or which break in the grips shall be discarded and retests made, unless such failures constitute a variable whose effect is being studied.

(d) Results on specimens which deviate markedly from the mean value of all tests shall be rejected if the deviation of the doubtful value is more than five times the average deviation from the mean value obtained by excluding the doubtful result. Such doubtful results shall be discarded and retests made unless the degree of variation is a factor which is being studied (Note).

Note: For some materials whose properties vary considerably throughout the sheet, as many as 50 specimen widths from portions of the sheet shall be tested, if a reliable picture of properties is desired.

**SPEED OF TESTING:**

The rate of stressing shall be held between the limits of 15,000 and 65,000 psi per min. in all tests. After an appropriate specimen width has been selected (Test specimen b), the desired stressing rate shall be obtained by varying the loading rate until the ratio of loading rate to specimen cross-section is approximately 40,000 psi per min.

**PROCEDURE:**

(a) A specimen width shall be selected which will produce failure well within the load limit of the testing machine. If necessary, a few trial runs shall be made in order to make a proper selection.

(b) The loading rate shall be set at a value such that it equals the product of the standard stress rate (15,000 to 65,000 psi per min.) and the cross-section (in square inches) of the specimen (Note I).

Note I: Recommended widths and loading rates are shown in the following table:

Nominal Thick- ness of Film, <u>in.</u>	Recom- mended Width of Speci- men, <u>in.</u>	Recom- mended Rate of Loading, lb. per <u>min.</u>	Recom- mended Rate of Stress- ing, psi <u>per min.</u>
0.003	0.5	60	40,000
0.005	0.5	100	40,000
0.0075	0.5	150	40,000
0.010	0.5	200	40,000
0.0125	0.5	250	40,000
0.15	0.5	300	40,000
0.020	0.375	300	40,000

(c) The thickness and width of the specimen shall be measured, reading the thickness to the nearest 0.0001 in. or better, and the width to 0.001 in. or better, at several points along its length. The minimum cross-section found shall be recorded.

(d) The test specimen shall be placed in the grips of the testing machine, taking care to align the long axis of the specimen with an imaginary line joining the point of attachment of the grips to the machine. (Note 2) At least 1 in. of specimen shall be included in each grip. The grips shall be tightened evenly and firmly to the degree necessary to prevent unnecessary slipping of the specimen during the tests; adjustment with a torque wrench to 50 in.-lb. is recommended.

Note 2: Great care in alignment is necessary to prevent failure by tearing rather than by true tension; and tests should be watched closely to make sure that tearing failures do not occur.

(e) A light line shall be ruled with ink or crayon, across the specimen adjacent to the edge of either grip.

(f) The recording pen shall be adjusted so that it starts at the proper point of the load-extension chart.

(g) While the specimen is being stressed, the displacement of the line described in Paragraph (e) shall be followed with respect to the edge of the grip with divider points, or other suitable devices. The value of this displacement shall be recorded at failure. This value shall be called the "correction factor."

#### CALCULATIONS:

(a) Breaking Load shall be calculated by dividing the maximum load in pounds by the original width of the specimen in inches. The result shall be expressed in pounds per inch of width and reported to three significant figures. The thickness of the film shall always be stated to three significant figures (Note 1).

Note 1: This method of reporting is useful for very thin films (0.005 in. and less) for which breaking load may not be proportional to cross-sectional area and whose thickness may be difficult to determine with precision.

(b) Tensile Strength shall be calculated by dividing the maximum (or breaking) load in pounds by the original minimum cross-sectional area of the specimen in square inches. The result shall be expressed in pounds per square inch and reported to three significant figures.

(c) The corrected extension shall be calculated by doubling the value of the "correction factor" (Procedure g) and subtracting the result from the extension read from the chart.

(d) Percentage Elongation shall be calculated by dividing the corrected extension at the moment of rupture of the specimen by the original distance between grips and multiplying by one hundred. The percentage elongation shall be reported to two significant figures.

**REPORT:**

The report shall include the following:

(a) Complete identification of the material tested, including type, source, manufacturer's code numbers, form, principal dimensions, previous history, etc.

(b) Method of preparing test specimens.

(c) Thickness and width of test specimen.

(d) Conditioning procedure used.

(e) Atmospheric conditions in test room.

(f) Number of specimens tested.

(g) Rate of loading.

(h) Rate of stressing.

(i) Tensile strength or breaking load, average value and average deviation.

(j) Percentage elongation average value and average deviation.

**REMARKS:**

(a) For all practical purposes the "breaking load" is reported as pounds per linear inch. This result gives a clearer picture of the performance of the material rather than the actual "tensile strength."

(b) This test also gives results for elongation, therefore, as STM will not be written for such a test.

(c) The above written test is essentially ASTM D-882-46-T. Reference should be made to this test method for the description of (a) stress, strain data and (b) Elastic modulus.

## APPENDIX F

### Flexible Packaging Division

Number STM-97  
Issued 3-16-55

NAME:

WATER-VAPOR TRANSMISSION RATE

PURPOSE:

To determine the water-vapor transmission rate on films or laminates when samples are either flat or creased as required.

EQUIPMENT & MATERIALS:

- (1) Humidity Cabinet: General Foods Humidity Cabinet providing a relative humidity of 90 to 95 per cent at a temperature of 100°F + or - 1°F with no condensation either on the test dishes or in the space where they are placed. Air circulation over test dishes must be negligible.
- (2) Special W.V.T.R. Test Dishes, Dessicators, and Brass Template: Must conform to the standard size used in the standard General Foods Humidity Cabinet.
- (3) Calcium Chloride. (Anhydrous min. 96%  $\text{CaCl}_2$ )
- (4) Wax Mixture (60% amorphous, 40% crystalline)
- (5) Heating Equipment for Wax.
- (6) 20 Mesh Screen, Weights (6 lbs. per inch).
- (7) Two 6" x 10" Flat Rigid Plates and 50 Ml. Beaker.

SAMPLE PREPARATION:

Cut 5.75 inch circular templates. Make minimum of 3 plates, flat and/or creased, on each structure (a 20.63 sq. in. area is exposed to test by the dish).

The creased samples, where required, are prepared as follows:

(1) Make three equidistant parallel folds in each circle by alternating the direction of folding on each successive fold so that the apex of each is facing an opposite side of the sheet in accordion style.

(2) Crease the folded circle by placing it between two 6 by 10-inch flat rigid plates and applying a total weight of 36 lbs. (6 pounds per inch of length of fold) for one minute. The center of gravity of the weight must be over the center of the sample.

(3) Open the circle, turn it upside down, and repeat the folding and creasing-under-weight process, making the second series of three folds perpendicular to the original.

**PROCEDURE:**

(1) Fill a 50-ml. beaker with 8-mesh anhydrous calcium chloride (min. 96 per cent as  $\text{CaCl}_2$ ) and decant into a thoroughly cleaned test dish, spreading it evenly over the bottom surface.

(2) Place the test specimen over the calcium chloride concentric with the rim of the dish.

(3) Coat the edge of the brass template, which fits over the specimen, with vaseline, taking care that none is on the surface which will be in contact with the sample.

(4) Heat the wax mixture in a porcelain crucible to at least  $212^{\circ}\text{F}$ .

(5) Filter through a 20-mesh screen to remove any large pieces of foreign matter and then pour into the annular space between the template and the rim of the test dish, filling up this space approximately flush with the top of the template.

(6) Cool and then remove template after wax is sufficiently hardened. Care must be taken not to cool the dish so long as to make the wax hard and brittle.

(7) Insert the test dishes in humidity cabinet.

(8) After 24 hours (or the interval from the time the plates are completed until 9:00 the next morning), remove the dishes from the humidity cabinet and place on racks in desiccators for 4 hours.

(9) Remove from desiccator and weigh (include the balance rack in the weight).

(10) Replace in the humidity cabinet for a period of 68 hours, remove, cool as before, and reweigh.

When testing cellophane:

(1) Remove from cabinet after initial conditioning period, place plates on racks and let stand at room temperature 15 minutes before weighing.

(2) Duplicate this procedure after the 68 hour exposure in humidity cabinet. (Excessive desiccation causes the wax to "pull away" from the outer perimeter of the plate.)

#### RESULTS:

The unit of water-vapor transmission is expressed in the grams of moisture per 100 square inches of area per 24 hours. The results of three determinations are averaged and reported as the final result. If these are inconsistent, conduct a duplicate set of tests to determine whether the variance is due to error in the procedure or to lack of uniformity in the sample submitted. If error in procedure is indicated, average the results of the second set.

## APPENDIX G

### Flexible Packaging Division

Number STM- 23  
Issued 5-13-57

**NAME:**

GAS PERMEABILITY DETERMINATION

**PURPOSE:**

To determine the gas permeability of flexible packaging material.

**EQUIPMENT & MATERIALS:**

- (1) Aminco - Air permeability apparatus.
- (2) Electric timer.
- (3) 1/2" and 3/4" wrenches.
- (4) Template 5" diameter.
- (5) Brodie Solution - Obtained from American Instrument Co.
- (6) Sample Size - 5" plus or minus 1/16" diameter. At least 3, preferably 6, specimens to be tested per sample.

**PROCEDURE:**

**I. WATER BATH TEMPERATURE:**

The water bath is maintained at a temperature of 25°C. A constant flow of cold water is run into the overflow unit.

**II. MANOMETER SOLUTION:**

With both arms of the manometer opened to the atmosphere, check the liquid levels at different positions. If both the left and right arm do not read exactly the same at the different positions, it will be necessary to clean the manometer.

**III. PREPARATION OF SAMPLE CELLS:**

- (1) Place a test sample on the porous metal disk, which serves as a support for the film.

(2) Place the pressure side of the cell on the studs. This side has a 1/4" copper tubing connection.

(3) Tighten belts evenly and place cells in the corresponding numbered positions in the water bath resting on the support tray. The 1/4" copper tubing should point towards the valves on the cell manifold.

(4) Tighten the 1/4" tubing collar to the valve on the manifold using a 1/2" wrench. Be careful not to exert too much pressure.

(5) Connect the metal capillary tube to the manometer by securely tightening the knurled collar. Make sure the metal capillary tube is inserted in the orifice leading to the manometer.

(6) Open the manifold valves and the manometer stopcock (in vertical position).

#### IV. TEST FOR LEAKAGE:

(1) Apply gas pressure to the system and visually inspect the cells for gas leakage. The water level should be above the collar on the 1/4" copper tubing leading directly into the pressure side of the cell. Gas leakage will be more easily detected with the water bath stirrer turned off.

(2) If gas leakage is noted tighten connection at point of leakage.

(3) Check the leakage at the cell to manometer connections by closing the manometer stopcock (etched line up) and raising the level of the Brodie solution on the left side about 10 cm. higher than the right side. If the left side falls rapidly or gradually, a leak is present, which will necessitate tightening of the knurled collar.

V. OBTAINING THE DATA:

- (1) With the manometer stopcocks and manifold valves open adjust the gas pressure to 14.7 psi.
- (2) Set the manometer level at 29.0 cm.
- (3) Close the stopcocks (etched line up) and start the timer simultaneously. Some films may require a conditioning period. This will be evident if the rate of Brodie solution displacement per unit time is not consistent.
- (4) The difference in Brodie solution level in each manometer should not be more than 0.4 dm. at any one time. The level is adjusted by turning the screw on the rubber tubing at the lower end of the manometer support.
- (5) Manometer level readings are taken at reasonable time intervals with both time and reading recorded.

NOTE: The time interval for reading the manometer level will vary with the permeability rate of the film being tested. Highly permeable films will require more frequent readings. At least 5 readings should be taken in order to plot the results on graph paper.

VI. CONCLUSION OF TESTS:

- (1) Open stopcock on manometer first. This is important! If not opened when gas pressure is relieved, the Brodie solution will back up in the cell.
- (2) Turn off the gas pressure.
- (3) Open the tubing from the cell to the manifold valve to relieve the pressure.
- (4) Record barometric pressure and room temperature.

(5) Dry the cells thoroughly and identify each specimen according to cell position.

RESULTS:

(1) Plot the displacement values versus the corresponding time on rectangular coordinate graph paper.

(2) Draw a straight line from the zero, zero point through the average of all points plotted.

(3) From this line determine the displacement value corresponding to one hour. Use this figure as cm displacement per hour, in the final calculations of the permeability as described below.

(4) Calculate the permeability in terms of cubic centimeters of gas per 100 square inches of test sample, per 24 hours, at a 1 atmosphere pressure and 25°C. as follows:

$$\text{Permeability} = \frac{DPF}{K}$$

Where D = cm displacement per hour  
P = barometric pressure in mm Hg.  
K = 273 + Room Temperature in °C.  
F = Manometer Factor.

(5) The manometer Factor, F, for each of the manometers in current use are attached at the end of the test method.

REMARKS:

(1) REPLACEMENT OF MANOMETER:

Whenever a manometer is replaced, it will be necessary to determine the volume and factor to be used for each replacement. This is done by weighing mercury which occupies a measured length in the manometer.

(2) DETERMINATION OF MANOMETER VOLUME:

Fill the manometer with clean mercury and accurately weigh a measured quantity (measured

in cm on the arm of the manometer). Record the barometric pressure and room temperature, and calculate the average volume per cm of the manometer as follows (corrected for 25°C and 760 mm pressure).

$$(a) \text{ Volume of } (V_1) \text{ of Hg} = \frac{\text{wt. of Hg.}}{13.546}$$

$$(b) \text{ Corrected Volume } (V_2) = \frac{V_1 \times P \times 298}{T \times 760}$$

Where  $P$  = Barometric Pressure  
and  $T = 273 + \text{Room. Temperature in } ^\circ\text{C.}$

$$(c) \text{ Volume per cm} = \frac{\text{Corrected Volume } (V_2)}{\text{cm of Hg. weighed}}$$

$$= V_2/\text{cm}$$

This expression  $V_2/\text{cm}$  represents the average volume of the manometer per cm corrected for 25°C and 760 mm pressure, and should be about  $0.002 \text{ cm}^3/\text{cm}$ .

### (3) CALCULATION OF MANOMETER FACTOR:

The manometer factor  $F$ , is calculated by multiplying the average volume per cm ( $V_2/\text{cm}$ ) by 74.864. This quantity is obtained by simplifying the basic permeability equation as follows:

$$\text{Permeability} = \frac{D \times P_1 \times (V_2/\text{cm}) \times \text{No. of Hrs.} \times 100 \text{ in}^2 \times T_2}{K \times A \times P_2}$$

$$= \frac{D \times P_1 \times (V_2/\text{cm}) \times 74.864}{K}$$

Where  $D$  = cm displacement of Manometer fluid.  
 $P$  = Barometric Pressure (mm).  
 $K = 273 + \text{Room Temperature in } ^\circ\text{C.}$   
 $V_2/\text{cm}$  = Average Manometer Volume per cm.  
 No. of hrs. = 24 hrs. used as standard.  
 $T_2 = 273 + 25^\circ\text{C}$  used as standard.  
 $P_2 = 760 \text{ mm Hg.}$  used as standard.  
 $A$  = area of test sample.  
 and  $F = V_2/\text{cm} \times 74.864$ .

Manometer Factor for Manometers in use at #102  
Research & Development are:

<u>Manometer Number</u>	<u>Factor</u>
1	1.4973
2	1.2932
3	1.4784
4	1.2572
5	1.4929
6	1.6020

## APPENDIX H

### Flexible Packaging Division

Number STM-147  
Issued 6-12-59

**NAME:**

POUCH BURST STRENGTH

**PURPOSE:**

To determine seal failure on liquid and cook-in type pouch structures.

**EQUIPMENT & MATERIALS:**

- (1) Equipment arrangement shown in diagram (A).
- (2) Machine or hand fabricated pouch samples

**PROCEDURE:**

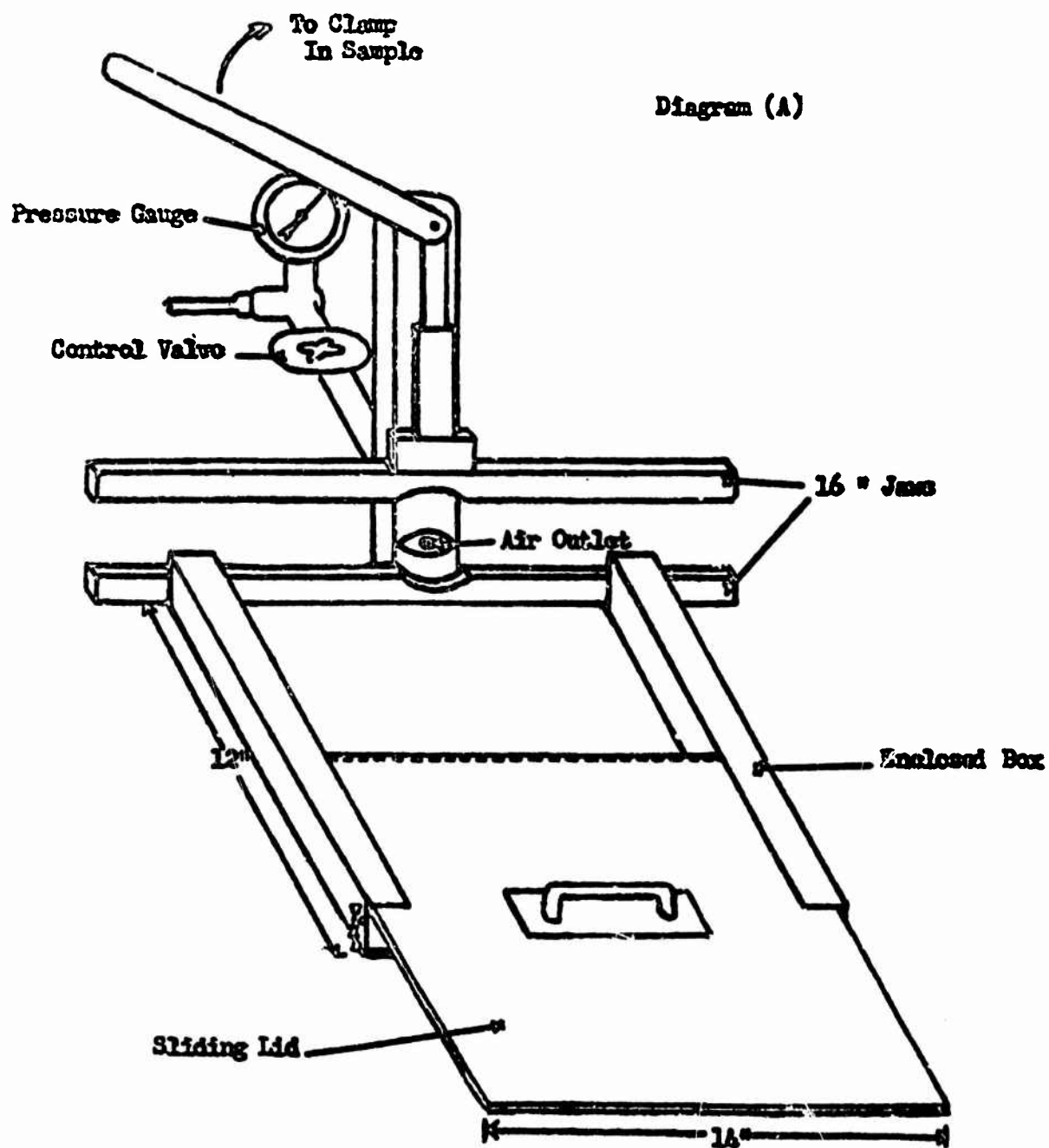
- (1) Adjust pressure gauge to 10 psi. (unless otherwise indicated).
- (2) Insert pouch lips around the air outlet and between the rubber covered jaws.
- (3) Clamp jaws tight on lips of pouch.
- (4) Release valve letting air into pouch gradually and hold for 30 seconds. Observe for bursting, and record time if a burst occurs.
- (5) Close valve and remove pouch.
- (6) If pouch does not burst, examine it for partial seal failure (defined as an irregular seal separation). If partial seal failure is noted, insert pouch again into the testing apparatus and check for an additional min.

**RESULTS:**

Report pouch burst failures and partial seal failures, and give their approximate locations. Also report the time for failure.

**NOTES:**

- (1) See product specification for requirements.
- (2) Product specifications will also include any pressure and/or time variations from the test above.
- (3) Do not use pouches which extend beyond the dimensions of the enclosed box.



## APPENDIX I

### Flexible Packaging Division

Number STM-152A  
Issued 5-20-60

- NAME:** IMPACT FATIGUE (IMPACT FATIGUE TESTER)
- PURPOSE:** To determine the impact fatigue resistance of pouches for packaging liquid products.
- EQUIPMENT & MATERIALS:**
- (1) Impact fatigue tester.
  - (2) Pouch test samples.
- PROCEDURE:**
- (1) Fill test pouch with water in the ratio of one ounce of water for every three (3) square inches of pouch inside dimension face area. (If the inside face area is 60 square inches, 20 ounces of water is used.) Then expel as much air as possible from the pouch and heat seal across the top of the pouch for closure.
  - (2) Place the filled pouch on the anvil.
  - (3) The plate is then weighed as specified in the Product Specification and released to drop on the pouch. The total plate weight, height of drop, and the minimum number of drops required without failure are as specified in the Product Specification.
- Note: A normal plate loading is one ounce per square inch of inside pouch face area.
- RESULTS:**
- (1) Report samples tested as passing or failing (any leakage of water from the pouch), giving the plate weight, height of drop, number of drops without failure, and the inside dimensional face area of the sample tested.
  - (2) Report position and extent of failures.

## APPENDIX J

### Flexible Packaging Division

Number STM-179  
Issued 2-25-65

- NAME:** PER CENT OXYGEN CONTENT OF FLEXIBLE POUCHES
- PURPOSE:** To determine the percentage of oxygen present in the headspace of a flexible pouch.
- EQUIPMENT & MATERIALS:**
- (1) Aerograph gas chromatograph manual temperature programmer (Model A-90-P) incorporating a 5A molecular sieve, 30-60 mesh. (Wilkens Instrument & Research, Walnut Creek, California)
  - (2) Recorder Model SR (E. H. Sargent & Company, Chicago, Illinois).
  - (3) Gas tight syringe, Teflon tipped 1 ML capacity desirable (Wilkens Instrument & Research, Walnut Creek, California).
  - (4) 25 ga. hypodermic needle.
  - (5) Prepurified nitrogen gas.
  - (6) Helium gas.
- PROCEDURE:**
- (1) Turn on and set helium carrier gas pressure to 50 psi.
  - (2) Check carrier gas flow into aerograph by means of the bubble flow meter located on the right side of the unit. Squeeze the rubber reservoir gently until the merging gas forms ascending bubbles in the flow meter. Measure the length of time it takes the bubble to rise from the 10 to 0 mark. Normal gas flow would take about 6 seconds.
  - (3) After the gas flow has been measured, turn the filament switch to the "On" position. It is

extremely important that carrier gas is flowing through the unit while the filament switch is on, otherwise damage to the filaments will occur.

(4) Turn recorder switch to standby.

(5) An instrument warm up period of 45-60 minutes should be observed.

(6) After the instrument warm up turn recorder from standby to drive position.

(7) Set recorder pen to 10ML on graph paper by adjusting fine tuning knob on the aerograph. This is the zero point from which all peak samples must be measured.

(8) For purposes to be explained later it is necessary to run an air sample prior to checking pouch headspace. Turn attenuator setting to 2 and readjust fine tuner position on recorder graph to 10.

(9) Inject .1 ML air sample into rubber diaphragm injector port. The first peak to appear on this sample will be oxygen. After the first peak turn the attenuator setting to 4 and adjust fine tuning so the recorder pen remains on 10. The second and last peak that will appear will be nitrogen. The total lapse of time from injection till the sample has cleared the system is about 3-4 minutes. Measure both peaks in ML and record for later use in determining oxygen content.

(10) For pouch headspace gas sampling attenuator settings will vary. On a presumed low oxygen content sample the oxygen peak attenuator setting would be 1 and the nitrogen 8 or 16 for a .3 ML sample injection. For higher oxygen content pouch samples it is recommended that the same attenuator settings and sampling amount that was used for the air sample be employed.

(11) Oxygen and nitrogen peaks will appear at the same point from the injection as did the pure air sample. Other peaks may appear, but should be disregarded for determination of oxygen per cent.

(12) Sampling of pouch headspace can now begin. Rinse the gas tight syringe prior to every sample taken by slowly drawing and expelling pure nitrogen from the syringe several times.

(13) Empty the nitrogen filled syringe just prior to puncturing the pouch sample to be tested. A slight hand pressure applied on the pouch will confine the headspace gas to one area for easier and accurate sample emission to the syringe.

(14) Remove, if possible, a sample of approximately .5 ML from the pouch and slowly expel .2-.4 ML prior to injection into the aerograph injector port. Attenuator settings are to be set as outlined in (10). Measure both peaks and attenuator settings.

(15) Pouch headspace oxygen per cent determinations can now be established per instructions set forth in calculation section of this STM.

(16) Upon termination of sampling it is important to secure the equipment in this order:

- (a) Filament switch off.
- (b) Recorder switch off.
- (c) Close helium and nitrogen main valves.
- (d) Relieve pressure on helium and nitrogen gauges.

CALCULATION:

(1) Pure air sample

$$\frac{N_2}{O_2} = \frac{78.1}{21.9} = X$$

21.9 = Oxygen content in air  
78.1 = Nitrogen etc. in air  
N<sub>2</sub> = Attenuator setting x Nitrogen Peak  
O<sub>2</sub> = Attenuator setting x Oxygen Peak

(2) Pouch test sample

$$\frac{N_2}{O_2} = \frac{100}{(X) + 1} = \text{Per cent oxygen content in pouch.}$$

OPERATIONAL DATA OF AEROGRAH MODEL A-90-P

<u>Unit</u>	<u>Temperature</u>	<u>Setting</u>	<u>Switch</u>
Injector	30 degrees C	5	Left on
Detector	50 degrees C	17	Left on
Collector	42 degrees C	-	-
Column	50 degrees C	-	-
Furnace & Fan	-	15	Left on
Filament Current	-	240 Milliamperes	Left on

## APPENDIX K

### Flexible Packaging Division

Number STM-180  
Issued 2-25-65

NAME: HEADSPACE GAS VOLUME DETERMINATION IN A FLEXIBLE POUCH

PURPOSE: To measure the volume of gas headspace in a flexible pouch by displacing water in a graduated cylinder.

EQUIPMENT:

- (1) Graduate, size would depend on pouch to be tested.
- (2) Funnel.
- (3) Deep sink or water bath.

PROCEDURE:

- (1) Fill sink with water.
- (2) Insert funnel into graduate and submerge under water.
- (3) Invert graduate so as bottom is above water. The graduate should be free of any air. If using a sink, be certain the drain plug is secure so as not to let any air gain entrance.
- (4) Take pouch to be tested and notch the seal area to insure easy opening.
- (5) Immerse the pouch under the funnel and tear open.
- (6) The water in the graduate will be replaced by the gas in the pouch and the total volume of gas headspace in the pouch can be easily read on the calibrated graduate.

## APPENDIX L

### Flexible Packaging Division

Number STM-181  
Issued 2-25-65

NAME: VACUUM DETERMINATION OF FLEXIBLE POUCHES

PURPOSE: To determine the amount of vacuum maintained in a flexible pouch.

EQUIPMENT:

- (1) Vacuum desiccator.
- (2) Vacuum pump.
- (3) Vacuum pressure gauge (MM. or inches).
- (4) Metering valve to control vacuum.
- (5) Non-collapsible tubing.

PROCEDURE:

- (1) Use the non-collapsible tubing to interconnect in line the desiccator, pressure gauge, metering valve and vacuum source.
- (2) Place the pouch to be tested inside the desiccator and secure lid.
- (3) Secure metering valve.
- (4) Turn vacuum pump on.
- (5) Regulate the metering valve so as to evacuate the desiccator slowly as possible.
- (6) Close visual inspection of the pouch during the evacuation of air is important to detect any movement of the pouch structure.

REPORT:

- (1) The vacuum inside the pouch will be equal to at whatever point during the evacuation pouch movement is evidenced. Coinciding this movement with the reading on the vacuum pressure gauge will tell in inches or millimeters the vacuum maintained inside the pouch.

(2) If no pouch movement is experienced after full line vacuum has been employed the pouch should be reported to have exceeded maximum vacuum obtainable whatever this may be.

## APPENDIX M

### Flexible Packaging Division

Number \_\_\_\_\_  
Issued \_\_\_\_\_

#### Toxicity Testing - Irradiated Products

##### POUCHES

- Pouches:
- (a) Cut off top seal (flamed scissors)
  - (b) Sniff test
  - (c) 20 gm sample removed (flamed forceps and scalpel) to sterile tared Waring Blendor cup.  
[Composites made from replicate pouches within a pouch variable.]  
Pouches are immediately resealed on a Sentinel Impulse sealer.
  - (d) Sterile water added to make 1:5 dilution
  - (e) Blended - 3 min.
  - (f) 40-50 ml homogenate to sterile screw cap tube "retained sample." Stored 38°F.
  - (g) 10-15 ml homogenate to sterile centrifuge tube (13 x 100 mm); centrifuged, 30 min. @ approx. 4000 rpm.
  - (h) Mouse injection:  
0.5 ml supernate (g) intraperitoneally injected into each of 5 Swiss-Webster albino mice.  
[Two sterile syringes used per sample, inoculating 3 mice with one and 2 mice with the other.]
  - (i) Mice housed in groups of 5 per cage.
  - (j) Mice examined for signs of toxicity and mortality at 4, 24, 48 and 72 hours following injections.  
Samples showing one or more deaths per group of 5 mice shall be retested, using supernate from the appropriate retained sample. Concurrently, mouse protection tests, using polyvalent antiserum, will be made (0.5 ml supernate + 0.25 ml antiserum to each of 2 mice).

One or more deaths in the second group of mice shall cause the sample group to be rejected.

- Cans:
- (a) Clean; open aseptically (bacteriological can opener)
  - (b) Sniff test
  - (c) 50 gm sample removed (flamed forceps and scalpel) to sterile tared Waring Blendor cup.  
Composites made of two or four cans to be used in organoleptic testing.  
Cans placed in 1 mil polyethylene bags (tied).
  - (d) - (j) As under "Pouches," above.

## APPENDIX N

### Flexible Packaging Division

Number AMXRE-FRP  
Issued 12-1-64

#### SUBJECT:

#### DEVELOPMENT OF BONELESS CHICKEN ROLLS

The previous method of cooking half breast and whole thighs in the steam chest was acceptable for metal cans. However, this method of preparation was not suitable for flexible packaging. The chicken breast varied in the degree of cooking because of the irregular size and weight. These breasts could not be put into flexible packages conveniently after cooking. The present work was designed to overcome these deficiencies by development of a boneless chicken roll suitable for metal cans and flexible packages.

#### EXPERIMENTAL:

The general procedure is that boneless skinned chicken breast is mixed with the emulsified breast, water and salt and stuffed in Visking Casings and cooked in the autoclave to 185°F and then cooled. The detail steps are as follows:

(1) Raw Material: The preparation of chicken for processing should include the following steps:

Fresh, ice packed, boneless chicken breasts weighing 7 to 8 ounces each should be obtained from local sources and should not be more than four days past kill. After receiving the chicken breasts, the skin, excess fat and bone should be removed. A loss of approximately 6-8% will take place during this operation. During this operation, ice should be added with the prepared breasts to maintain the temperature as near 32°F as possible.

(2) Preparation: In preparation of the breast for stuffing, a formulation of 350 gm of breast, 500 ml of tap water and 35 gm of salt was placed in a 1 gallon Waring Blendor container and blended for 20 seconds on medium speed. The emulsified mixture was mixed with 8 pounds of the chicken breast in a large pan. The resulting mixture was then stuffed in casings.

This mixture is made up of the following proportion:

80.4% Whole Boneless Skinned Chicken Breast  
7.7% Emulsified Chicken Breast  
11.1% Water  
.8% Salt

Other formulations tried included 3, 6, 9, and 12 per cent emulsified breast, 0, 1, and 2 per cent added salt and 5, 10, 15, and 20 per cent added water. In addition, the emulsified chicken breast was added in amounts of 0, 5, 10, 15 and 20 per cent to the whole chicken breast. The above formulation was determined to be most suitable by a technical panel. This formulation gave a product with a minimum of air spaces, good binding qualities, no discoloration and an acceptability score of 7 both before and after irradiation.

(3) Stuffing: The stuffing of the boneless chicken breast was accomplished by using Visking Casing, the Smeco Meat Press and the Globe "U" Clipper. Number 7 Visking Fibrous Casing are clipped on one end after folding and then placed in 100°F water until used. The casings are pulled over the 3.5 inch horn of the Smeco Meat Press as far as possible. The meat press should be set at 60 lbs. pressure for stuffing. The chicken breast should be placed in the meat press and forced out slowly into the casings. Pressure should be applied to the casing on the horn during filling to insure maximum extension of the casings. After stuffing in the casing, it should be removed to the Globe "U" Clipper where pressure is applied to the casing by pulling on the end. The casing should be stuffed to a circumference of 14.5 inches and a length of approximately 17 inches.

(4) Cooking: Cooking should be done in the autoclave. The stuffed chicken rolls should be placed on the basket of the autoclave so that the rolls are not touching. Thermocouples can be inserted into the center of the rolls to follow the temperature rise during cooking. The autoclave should be set at 106°C (223°F) and the timer set for 107 minutes. This time is approximate for cooking to 185°F starting from approximately 45-50°F. Cooking of rolls of this diameter have a temperature rise internally of 1.7°F per minute after an initial lag of 15 minutes. The surface temperature of the rolls will approach the temperature of the autoclave within the first 10-15 minutes. A graph of temperatures during cooking and cooling of the rolls is included.

(5) Cooling: Immediately upon removal from the autoclave, the casings should be retightened to the desired diameter on the Globe "U" Clipper. The excess casing should be cut off and the product placed in a 35°F blast cooler for not less than 4 hours. After cooling, the product can either be cut into uniform slices or cut for insertion into metal cans.

The following times and temperatures were recorded. The minutes given are for the time the internal temperature was at or above the given temperature during cooking and cooling.

<u>Temperature (°F)</u>	<u>Time (Min.)</u>
130	100
140	87
150	73
160	57
170	42
175	34
180	25
183	16
185	3

The above temperatures are an average of 10 individual rolls. The standard deviation at 185°F was  $\pm 2.8$  F. This does not include any adjustment for thermocouple location within a roll.

The cooling of the rolls follows a line similar to the heating when the temperature difference is more than 90°F. The enclosed graph shows the cooling of the product in a 35°F blast cooler. A shrink of approximately 34% from the initial weight of roll is encountered.

(6) Packaging: After cooling, the casing is removed and product is ready for packaging. For metal cans, the rolls can be cut to the desired length and inserted into the No. 2 or No. 3 cans. At least 3/4 inch of headspace should be allowed and the cans closed under full vacuum.

For flexible packages, the product should be sliced 1/2 inch thick on the Globe Slicing Machine. The slices should then be trimmed to 2-7/8 by 4 inches. Using the filling tube made by Continental Can Company, the sliced product can be placed in the 4-1/2 by 7 inch pouches. The packaged product is then sealed under 25 inches of vacuum on the Flexvac machine.

(7) Evaluation: A technical panel evaluated the product packed in metal cans before and after irradiation. The sliced chicken was heated in a 350°F oven for 20 minutes. The sliced product scored 7 on a 9 point Hedonic scale with practically no irradiation flavor.

The sliced chicken held together during reheating and could be served as whole slices. The texture was that of chunk chicken pieces. The product could be consumed as cold chicken or broken up for use in salads, chicken alfredo, etc.

**SUMMARY:**

This work resulted in a product which can be used in both metal cans and in flexible packages. A more uniform dose distribution is obtained in the metal cans because of the nearly solid pack throughout plus more weight per unit volume within the can. A portion control product can be obtained by processing the chicken in this manner in both flexible and metal containers. This product when cooked to 185°F with the added salt can be consumed as cold or reheated. The flexible pouch with a 4-ounce serving could be used in individual rations.

The final sliced chicken roll does not look like the original breast or thighs which may be a slight disadvantage. The product does have whole chunks of chicken when sliced and has better texture than a ground product.

**FUTURE WORK:**

The following suggestions are made for additional work to be considered in this area. For flexible packages, a spring tension load pan should be considered. This would eliminate the cutting of the round rolls to fit the rectangular pouches.

The use of skin as the binding agent should be investigated. A Griffith Mince Master emulsifier may be used to emulsify the skin so that the discoloration observed in this study when skin was used may be eliminated. A more uniformly ground product should be obtained with this type of machine.

More extensive work should be conducted in the area of water, salt and chicken quantities to develop more data concerning the function of each material. A more satisfactory binder may be developed in this manner.

Observing the time-temperature relationship during the cooking and cooling of the product, it may be possible to lower the final internal temperature because of the time at each temperature. The necessary enzymatic kill may be obtained at 175°F. This would result in less shrink and a juicier product.

A similar procedure should be investigated using thighs. This process should be applicable to thighs as well as breast.

MELVIN O. WARNECKE  
1st Lt.           Cmlr  
Food Laboratory

**Copies furnished:**

Dr. E. Wierbicki  
Dr. C. K. Wadsworth  
Mr. J. Killoran  
Dr. F. Heiligman  
Lt. M. O. Warnecke

## APPENDIX O

### Metal Container Division

Number STM-1  
Issued 7-1-64

### PROCEDURE FOR ORGANOLEPTIC STUDIES ON TEST PACKS

Informal "in-package" sniff tests were conducted by one person at the time of the initial opening of the pouches for microbiological tests. Formal organoleptic panel tests were conducted following the completion of 72 hour microbiological tests.

The organoleptic panel consisted of ten persons immunized for botulinum studies. Samples from only one storage temperature were evaluated at any one session. The order of samples served was randomized in order to minimize "fatigue" and "first-sample" effects. The meat was diced and served at room temperature in ruby red glasses with watch glass covers. Bacon samples were baked for ten minutes at 375°F. Samples were evaluated at one, three, six and twelve months.

The Multiple Sample Difference Method (a modified paired-comparisons test, developed at Continental Can Company) was followed. The following questionnaires were used.

This outline covers both the "In-Package" Odor Tests (to be run at the time sample material is removed from each pouch for mouse testing) and the formal panel flavor tests (including both odor and taste) to be run upon the completion of the 72-hour mouse tests. It includes the following information:

I. "In-Package" Odor Tests

- A. Schedule
- B. General Instructions
- C. Questionnaire

II. Panel Flavor Tests

- A. Schedule
- B. General Instructions
- C. Questionnaire
- D. Preparation of Samples

I. "IN-PACKAGE" ODOR TESTS

A. SCHEDULE

<u>PRODUCT</u>	<u>DATE PACKED</u>	<u>MONTHS STORED (1)</u>				
		<u>ONE</u>	<u>THREE</u>	<u>SIX</u>	<u>NINE</u>	<u>TWELVE</u>
Ham	10/27/64	11/16/64	1/26/65	4/26/65	7/27/65	10/25/65
Chicken	10/29/64	11/17/64	1/25/65	4/27/65	7/26/65	10/28/65
Bacon	10/28/64	11/24/64(2)	2/1/65	5/8/65	8/2/65	11/1/65

(1) Includes all three temperatures variables

(2) Date may have to be changed due to complication of holiday week

B. GENERAL INSTRUCTIONS

Each pouch should be sniffed immediately upon opening. The attached questionnaire should be filled in as the pouches are opened.

"IN-PACKAGE" ODOR TEST

IRRADIATED MEAT

<u>STORAGE CONDITIONS</u>	<u>S A M P L E S</u>											
	<u>75°F-50% R.H.</u>				<u>100°F-10% R.H.</u>				<u>100°F-90% R.H.</u>			
<u>POUCH:</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>

INTENSITY OF ODOR:

(Check one for  
each sample)

- a. Trace
- b. Slight
- c. Moderate
- d. Strong
- e. Extremely  
strong

OVERALL QUALITY OF ODOR:

(Check one for each  
sample)

- a. Appealing
- b. Neither  
appealing nor  
unappealing
- c. Unappealing

CHARACTER OF ODOR:

(Describe if  
possible)

## II. PANEL FLAVOR TESTS

### A. SCHEDULE

PRODUCT	DATE		MONTHS STORED*			
	PACKED	ONE (3-4 Weeks)	THREE	SIX	NINE--OR--TWELVE	
Ham	10/27/64	11/19/64	1/29/65	4/29/65	7/30/65	10/28/65
Chicken	10/29/64	11/20/65	1/28/65	4/30/65	7/29/65	10/29/65
Bacon	10/28/64	11/27/64**	2/ 4/65	5/ 6/65	8/ 5/65	11/ 4/65

\* Includes all temperature variables.

\*\* Date may have to be changed due to complication of holiday week.

### B. PANELISTS

The following persons should participate in all examinations. Since there is a limited number of persons who can participate in this test, it is extremely important that the same persons participate in all tests. The following persons have been immunized and should participate in these tests.

1. J. Boltz
2. J. Bock
3. J. Folinazzo
4. D. Huber
5. R. Juritz
6. D. Maunder
7. J. Novak
8. W. Robertson
9. C. Schmidt
10. W. Sogner

Other persons who previously demonstrated ability to discriminate with irradiated food odors may be used in the panels for odor only and as a supplement to the immunized panel if sufficient sample is available.

The following is a list of these persons:

Glenn Dean

M. Fuhrman

J. Houston

F. Kraus

W. Sessler

T. Steiskal

#### C. GENERAL INSTRUCTIONS

The following order of testing was observed in all examinations.

Test 1	10:00 A.M.	75°F, 50% RH Samples
Test 2	11:30 A.M.	100°F, 10% RH Samples
Test 3	3:15 P.M.	100°F, 90% RH Samples

Samples from more than one storage temperature should not be evaluated in the same session. One product only should be evaluated in any one test.

(Chicken and ham should never be served in the same test.)

The Multiple Sample Difference Test will be used. (See attached questionnaire.)

The order of samples served within each test should be randomized, regardless of the position of the blind standard.

All samples should be served at room temperature in ruby red glasses with glass covers. The taster should have a clean spoon for each sample. The following instructions should be posted in each booth:

#### IRRADIATED MEAT

Please concentrate on "differences" between these samples.

Carefully "sniff" all samples and compare them for odor before tasting any of them. When you have recorded your odor comments you may proceed with tasting the samples.

You may rinse your mouth between samples, if you wish--but, if you do, you must rinse between all samples.

The name of the particular product under test should appear on the questionnaire ("Ham," "Chicken," or "Bacon").

#### D. QUESTIONNAIRE

See the next page.

IRRADIATED FOOD

CHICKEN

NAME \_\_\_\_\_ DATE \_\_\_\_\_ TIME \_\_\_\_\_

DIFFERENCE TEST

Sample K is the standard, or reference, sample. Please rate the quality of K below.

Quality of K (Circle One)	Comment about K	
_____	Odor _____	Flavor _____
Excellent		
Good		
Fair		
Poor		

Please compare all other samples to K for odor and flavor differences. Test samples may or may not be different from the standard. Rate each test sample according to this difference scale.

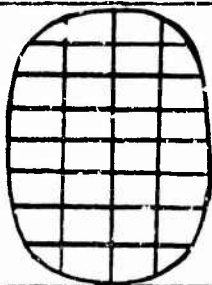
<u>Rating</u>	<u>Difference from Standard</u>
0	No difference
1	Very slight difference
2	Slight difference
3	Moderate difference
4	Large difference
5	Very large difference

Test Sample	Difference Rating		Comments		Quality of Samples as Compared to K (Check One)		
	Odor	Flavor	Odor	Flavor	Inferior	Equal	Superior

#### E. PREPARATION OF SAMPLES

##### Chicken

- a. Dice each chicken slice into 32 1/2-inch cubes as follows:



- b. Mix cubes from all slices of chicken for any one code at any one storage condition. (There should be four slices of chicken in all.) Blend cubes until they are well mixed.
- c. Serve each taster approximately five cubes.

##### Ham

Prepare as in chicken. Try to serve each taster approximately three lean cubes and two fatty cubes.

##### Bacon

- a. Separate lean-end slices from fatty-end slices. There should be about five of each in one pouch. Set aside the odd pieces which have been added to the pouch for weight.\* Keep each cube separate. Handle pieces of bacon carefully as they are thinly sliced. (\*Do not use the odd pieces unless it is necessary.)
- b. Prepare a set for each taster by placing one lean slice and one fatty slice from each cube in a marked position on a rack. Include two standard samples
- c. Bake the entire rack of samples at 400°F for ten minutes. Remove from oven and allow to cool (on rack). Carefully remove samples from rack and place in ruby red glasses. Cover.

CCC Form No. 6-40-3

**E. EVALUATION**

Data from the questionnaires should be recorded in the Flavor Panel  
Record Books. Questionnaires should then be forwarded for evaluation.

Unclassified  
Security Classification

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<i>(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)</i>		
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13. ABSTRACT		
<p>Flexible packaging laminates were developed and evaluated for use with prepackaged irradiation-sterilized ham, bacon and chicken. Laminate structures comprised poly (ethylene terephthalate) as the outside ply, aluminum foil as the middle ply, and heat sealable inside ply, namely film of polyolefin, polyamide or polyester. These laminates were evaluated for food compatibility, low-temperature resistance, and resistance to microbial and insect penetration. The flexible laminate with the polyamide as the food-contacting film was found satisfactory for use with prepackaged ham, chicken and bacon in various test environments over a one year storage. A flexing apparatus, developed to induce bacterial penetration through defects in flexible packages, was found capable of detecting leaks not visible to the naked eye. The three types of flexible laminates showed no negligible weight loss and no package rupture in shipping vibration tests performed as low as -180°C. Exposure of the flexible laminates to three types of boring insects over a three month period caused no complete penetration of the packages under study.</p>		

DD FORM 1473

REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

Unclassified  
Security Classification

Unclassified  
Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Packaging	8		6		6	
Radiation-sterilized	0					
Ham	9					
Bacon	9					
Chicken	9					
Flexible	0					
Laminated plastics	10		6		6	
Compatibility			7			
Food			7			
Resistance					7	
Temperature					7	
Microorganisms					7	
Insects					7	

Unclassified  
Security Classification